AD

TECHNICAL REPORT

70-16-FL

RECOVERY OF COMPRESSED DEHYDRATED FOODS

by

A. P. MacKenzie

and

B. J. Luyet

American Foundation

for

Biological Research

Madison, Wisconsin

Contract No: DAAG 17-67-C-0126

July 1969

UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760

Food Laboratory

FL-90

AD_____

TECHNICAL REPORT
70-16-FL

RECOVERY OF COMPRESSED DEHYDRATED FOODS

by

A. P. MacKenzie B. J. Luyet

American Foundation for Biological Research
Madison, Wisconsin

Contract No. DAAG 17-67-C-0126

Project reference:
1M624101D553

Series: FL-90

July 1969

Food Laboratory
U. S. ARMY NATICK LABORATORIES
Natick, Massachusetts 01760

FOREWORD

The logistic significance of reducing the weight and bulk of operational rations has long been recognized. In varying degrees, weight and bulk are major factors in the packaging, handling, storage and transportation of all military subsistence. These factors become critical in the design of a food packet for special missions on which a soldier must carry his entire food supply for periods up to eight days. For a great variety of foods freeze drying combines reliable preservation with maximum weight reduction and provides for rapid rehydration to yield products of superior acceptability. Freeze drying, however, does not result in a consequential decrease in bulk. Previous studies have demonstrated that most freeze dried foods, when properly plasticized, can be compressed to a volume ranging from one-third to one-twentieth of their initial volume. This compression can be achieved without a consequential amount of fragmentation and is reversible, in that on rehydration the product returns to its prefrozen size and shape with no apparent damage from the compression experience.

This investigation seeks to extend the knowledge of factors which influence the reversible compression of freeze dried foods and to develop procedures to assure the reversible compression of representative dehydrated foods. In conjunction with these objectives, this investigation also seeks to identify the structural components of mechanisms by which various foods achieve recovery from compression. A corollary to this latter study is expected to provide insight into the nature of compression damage which results in irreversibility.

This investigation was conducted at the American Foundation for Biological Research, Madison, Wisconsin 53704, under Project: 1M624101D553, Food Processing and Preservation Techniques. Dr. Alan P. MacKenzie served as Principal Investigator in association with Dr. B. J. Luyet, Director of the American Foundation for Biological Research. Dr. M. B. Farhoomand, and Messrs. C. A. Kroener, T. A. Kuster, A. R. Kutchera, J. E. Mundstock, G. R. Orndorff and D. H. Rasmussen assisted. The Project Officer for the US Army Natick Laboratories was Dr. Maxwell C. Brockmann. Dr. Karl R. Johnson served as Alternate Project Officer.

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

TABLE OF CONTENTS (Continued)

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

LIST OF PHOTOGRAPHS

LIST OF PHOTOGRAPHS (Continued)

LIST OF PHOTOGRAPHS (Continued)

LIST OF PHOTOGRAPHS (Continued)

LIST OF PHOTOGRAPHS (Continued)

LIST OF PHOTOGRAPHS (Continued)

LIST OF PHOTOGRAPHS (Continued)

ABSTRACT

Seven foods were frozen at various rates, freeze-dried in different ways and adjusted to different moisture contents by exposure to atmospheres of controlled R.H. The resultant materials were compressed, dried, and tested for their capacities to recover initial form and quality on rehydration.

It was observed that processing conditions insuring best recovery can be defined in terms of relative humidity to which a food is exposed prior to compression.

Freeze-dried foods of predetermined moisture content were produced by newly developed processes involving only the sublimation of ice and the direct desorption of a part of the water remaining unfrozen. These methods offered effective alternatives to the method by which fully freeze-dried materials are moistened by resorption prior to compression.

Compression in vacuum was successfully demonstrated. Similarly, freeze-drying, adjustment of the water content, compression, and final drying were realized in a single apparatus. These methods were each shown to possess special advantages.

Additional experiments were conducted on the compression of solvent-extracted foods. Freeze-dried, compressed, and restored foods were also examined by light and electron microscopic techniques. From these additional studies some indications were obtained of the mechanisms and factors contributing to restoration.

INTRODUCTION

This study was undertaken in an attempt:

(1) to develop procedures which assure full recovery of representative dehydrated foods from the effects of compression,

(2) to describe the functional components and mechanisms which contribute to this recovery,

(3) to provide an insight into the nature of those irreversible changes which preclude full recovery.

The work was conducted in a manner permitting a comparison of the known methods for the production of freeze-dehydrated compressed foods with a method developed during the years 1964 through 1966 at the American Foundation for Biological Research and designated "limited freeze-drying" (MacKenzie, 1965, 1967).

Previous studies showed that the capacities for "restoration" evidenced by freeze-dried, compressed foods depended principally on:

(1) water content and temperature of the sample during compression, the pressure employed and the duration of that pressure,

(2) the temperature/water content profiles during final drying and the maintenance of physical pressure during the final drying stage,

(3) conditions during storage (Lampi, et al., 1965; Ginette, 1966).

It seemed to us that the properties of the freeze-dried compressed foods should also depend on:

(1) the structure of the food after freezing. This is necessarily determined by the temperature profile history during freezing and the direction of freezing with respect to the orientation of organized biological structures; i.e. by the "mode of freezing."

(2) the way in which the temperature/water content profile changes during freeze-drying; i.e. by the physical form and manner of operation of the freeze-drying apparatus or equipment.

Thus we adopted a dual approach. On the one hand we undertook a study comprising the following operations:

(1) each food was frozen at each of two well-separated rates,

(2) two freeze-drying procedures were applied to each food, frozen each way,

(3) compression of each freeze-dried product was conducted at each of three levels of residual water content (r.w.c.) but only at room temperature,

(4) two types of compression procedure were investigated.

Each of the two compression procedures was employed to follow the form of freeze-drying to which it was better suited.

On the other hand we conducted supplementary experiments to aid in the understanding of the mechanisms operative during the restoration of original structure upon rehydration. These included:

(1) gross studies on freeze-dried, solvent-extracted, compressed materials,

(2) cytological studies on compressed and restored specimens, fixed, embedded and sectioned by light microscopy,

(3) ultrastructural studies of freeze-dried compressed foods in the scanning electron microscope.

Practical systems incorporating improvements resulting from studies completed under the various items outlined above were constructed and evaluated with reference to pilot-plant scale operation.

MATERIALS

The following materials were acquired for the purposes of the study: beef, cabbage, carrots, peaches, peas and shrimp.

Beef was purchased several days post-mortem in the form of 3 to 4-pound sirloin cuts, graded U.S. Choice, and selected for their leanness.

Cabbages of an undetermined variety were purchased locally. Heads weighed about 2 pounds each and were judged to be uniformly and firmly filled with crisp white tissues.

Red-Core Chatmay carrots were obtained from Libby, McNeil and Libby, Inc., Darien, Wisconsin, scrubbed, blanched, diced and inspected prior to delivery,

Early Frosty peas were obtained from Libby McNeil and Libby, Inc. They were delivered shelled, blanched, size-graded and inspected.

Gulf shrimp judged to yield a 26-35 regular count, flown live to Madison, were purchased when cooked, shelled and deveined.

METHODS

1. Preparation for Freezing.

(a) Raw beef. Sirloin was inspected for fiber direction and cut into slices 1 cm. thick in a room at 2°C. Portions of the slices free from fat and connective tissue were cut into 1 cm. cubes.

(b) Cooked beef. 1 cm. thick slices, prepared as described in (a) were wrapped in aluminum foil. A copper-constantan thermocouple was located in the center of the slice. The aluminum packets were individually immersed in gently boiling water, removed when the interior temperature of the slices reached 80°C. Portions of each slice free from fatty and connective tissues were cut into 1 cm. cubes.

(c) Cabbage. Outer leaves were stripped away to yield white interior tissues. These tissues were cut into shreds approximately 5 mm. wide.

(d) Carrots. Freshly cubed carrots were rinsed once in cold water, cooked in 2-lb. batches in wire-mesh baskets immersed in minimum volumes of slightly salted water for periods of 20 minutes, drained and cooled by immersion in water at 12 to 15°C. Baskets were shaken several times during cooking to aid production of uniformly cooked material.

(3) Peaches were blanched for one minute each in boiling water, skinned, pitted, and cut into conventionally wedge-shaped slices having a thickness of 12 to 15 mm. measured across the outside of the middle of the wedge.

(f) Peas were rinsed once in cold water, cooked in 2-lb. batches for 8 to 10 minutes in lightly salted, boiling water, drained and cooled in water 12 to 15°C.

(g) Shrimp. It was only necessary to soak the shrimp for 10 minutes in cold water at 15°C.

2. Freezing.

Each of the seven foods was subjected to the same slow freezing procedure and to one of three rapid freezing processes. The greatest care was taken to prevent the foods from undergoing any surface drying following preparation and prior to freezing.

Slow freezing. Foods were spread one layer thick (cubes of beef, peach slices, whole shrimp) or in multilayers 3 to 4 cm. thick (shredded cabbage, carrot cubes, peas) on Teflon-coated aluminum trays and placed on wooden tables in a cold room maintained at -40±2°C. A forced air circulation was not maintained in the room.

Prior to freezing, 3-mil. copper-constantan thermocouples were placed at the centers of several pieces of each type of food and temperatures were recorded during freezing.

Rapid freezing. First method : This method represents an attempt to duplicate the commercial efforts to freeze foods in a rapidly moving stream of dry nitrogen gas at or near -196°C. For this purpose the apparatus shown in Fig. 1 was constructed and operated successfully. Single layers of peaches (1 piece in each batch contained a thermocouple) were processed in this manner until approximately 20 pounds of frozen fruit were accumulated. The method proved to be too time consuming and was not used for the other foods.

Second Method: A compromise means of rapid freezing in liquid nitrogen was devised in which baskets full of peas were dipped briefly and repeatedly into liquid nitrogen, with shaking, and permanently immersed only when each pea was partially frozen. Thirty pounds of cooked peas were frozen in this manner. This method also had disadvantages and was used only to freeze peas.

Third method: More practical for handling 10-to 50-pound quantities than liquid nitrogen, solid carbon dioxide was also used for rapid cooling. Powdered dry ice was sifted into a Styrofoam-walled container by one person while a second person scattered pieces of food on the bed so formed. Rates of addition were adjusted to about 2 lbs. of dry ice per pound of foodstuff, yielding essentially separate freezing of each piece of food. Dry ice and food were separated after 30 minutes with the aid of sieves. The method was employed with complete success in the rapid freezing of raw beef, cooked beef, cabbage, carrots and shrimp. As in the other freezing experiments thermocouples were located in selected food pieces and yielded typical cooling curves. Up to 50 pounds of food could be frozen this way in a single insulated container.

3. Frozen Storage.

All the foods were placed after freezing in polyethylene bags, five to ten pounds to a bag, and stored in large protective containers in a cold room at -40±2°C.

4. Freeze-Drying and Associated Operations — Physical Compression/Resorption Studies.

(a) Compression of overdried, remoistened material

(i) Freeze-drying at room temperature was conducted in the apparatus shown in Figure 2. Approximately 100 g. of material were loaded into each of four 2-liter flasks in a room at -40°C. (one food, frozen at one of two rates, per flask). The flasks were attached to the apparatus by means of silicone rubber o-rings and special clamps. Dry ice was added to the condenser and a vacuum was drawn on the sample chamber/condenser assembly. Freeze-drying was immediately initiated, proceeding, as in a commercial apparatus, at such ice-interface temperatures as might result from the necessary balancing of heat and mass transfer. Judged by the feel of the 2-liter flasks to the hand, the freeze-drying of these 100-g. quantities was completed in 16 hours or less.

Vacuum was broken with argon via the stopcock attached to the bottom of the condenser chamber. Flasks were immediately removed from the apparatus and capped with airtight plastic lids.

(ii) Humidification. Dry materials were exposed to moist conditions in glass vacuum desiccators containing various concentrations of sulphuric acid in water. Each desiccator was, in turn, connected to a vacuum gauge as shown in Figure 4. In this way the relative humidity established in each desiccator was quickly checked. Division of the pressure reading by the pressure exerted by water at the same temperature yielded the relative humidity directly.

Fourteen different types of freeze-dried sample (seven foods, two freezing rates — several grams of each) were placed in each of seven desiccators set up to provide at 25°C., 20, 30, 40, 50, 60, 70 and 80% R.H., respectively. The desiccators were kept evacuated for a week. Once a day the sulphuric acid solutions in each desiccator was stirred magnetically, to avoid a possible "layering", without interruption of the vacuum. Desiccators were also checked daily for leaks. A period of equilibration of one week was employed because, at the start of the work, it was not known how quickly moisture contents attained equilibrium values in the absence of air. Ratios of food to sulphuric acid solution were such that acid concentrations did not change appreciably during the course of the equilibrations.

(iii) Compression/restoration tests. The vacuum in each of the seven desiccators was released at the same time and the fourteen series of foods (seven foods, two freezing rates) taken one at a time for compression studies. Individual pieces of each food exposed to each of seven different relative humidities (and hence brought to seven different moisture contents) were subjected, between two small aluminum discs, to various pressures. The foods were removed from the press and rehydrated. Each sample's behavior was noted with reference to sample orientation, pressure, duration of compression, and method used to permit restoration (flotation vs. forced immersion; hot water vs. cold water). Foods were examined one at a time, to evaluate the effects of different humidities.

(b) Compression of materials subjected to limited freeze-drying.

NOTE: Limited freeze-drying refers to a process developed between 1964 and 1967 by Dr. A. P. MacKenzie at the American Foundation for Biological Research. In this process the sample is placed in a chamber maintained at a given low temperature (T_1). The condenser is maintained at a somewhat lower temperature (T_2) generally 5 to 20 degrees lower than T_1. The limited freeze-drying process is conducted in such a way that the total pressure in the system tends to that of the vapor pressure of ice at the temperature of the condenser. Such other gases as may evolve from the sample or leak into the apparatus are subjected to continued pumping and do not contribute to the final equilibrium vapor pressure over the sample. It is possible by this means to freeze-dry to moisture contents predetermined by the suitable choice of T_1 and T_2. The method has been subjected to numerous successful tests in the laboratory.

(i) Limited freeze-drying with stepwise decrease in condenser temperature. Food was prepared in quantities sufficient for preliminary compression/restoration behavior tests in the apparatus for limited freeze-drying (Figure 5). Sample chamber and condenser temperatures were separately controlled according to the method described by Luyet and Rapatz (1957). Samples were processed two at a time (one food, two freezing rates) in five separate preparatory experiments. Due to their tendency to undergo structural collapse, two foods, peaches and cabbage, were not processed in this apparatus.

Limited freeze-drying was in each case carried to completion at a sample chamber temperature of -10°C. and a relative humidity (in the sample chamber, at -10°C.) of 60%. The termination of desorption following the completion of sublimation was judged by the result of intermittent closure of the stopcock located between the sample chamber and the condenser (the method of vapor pressure rise to equilibrium or v. p. r. t. e.). Five to ten grams of each food were then withdrawn from the apparatus. The condenser was then lowered a predetermined number of degrees, causing the desorption of water to be resumed (due to the reduction of the R.H. in the sample chamber resulting from a decrease in condenser temperature). When v.p.r.t.e. showed the establishment of a second equilibrium, the apparatus was reopened and further quantities of material withdrawn.

Samples were, in this way, prepared by desorption to relative humidities in the sample chamber, at -10°C., of 60, 50, 40, 30, 25 and 10%. Samples removed from the sample chamber at -10°C. were in each case warmed to room temperature under argon in sealed chambers to await compression.

(ii) Compression/restoration tests. These tests were performed in an identical manner to those on resorbed materials. See 4, (a), (iii). The behavior of each food at six moisture contents was securely established by repeated experiment before the next food was examined.

5. Freeze-Drying and Associated Operations — Quantitative Data.

(a) Production and subsequent processing of overdried materials.

(i) Determination of the velocities of freeze-drying. The first object of this series of experiments was the provision of data to permit a comparison between the rates at which foods could best be prepared for compression by freeze-drying "at room temperature" and by "limited freeze-drying". Thus sample size and sample chamber geometry were chosen to permit as unbiased a comparison as possible. The apparatus used consists of a freeze-drying apparatus comprising a vacuum system and a condenser separated from the sample chamber by a space incorporating a continuously recording balance (Figure 6). To make a run, a wire mesh basket was loaded with approximately 20 g. of material in a room at -40°C., carried in an insulated vessel to the apparatus and suspended from the balance beam. Thermocouple leads were connected. The sample chamber was attached to the balance chamber and the balance quickly tared electrically to provide a full scale deflection on a 1 mv. stripchart recorder. The system was then rapidly evacuated with the result that freeze-drying was initiated in a matter of seconds. Such recorder chart speeds, range settings, etc., were used as might permit the most accurate reconstruction of the weight/time curves, tangents to which afforded the measurements of the freeze-drying rates (in terms, e.g., of the weight of water lost per unit initial weight).

(ii) Resorption isotherms. Quantities of each of the fourteen test materials were prepared by freeze-drying at room temperature as described in Section 4, (a), (i), divided and placed in preweighed bottles. These bottles, with their contents, were then transferred to desiccators filled with freshly baked solid desiccant (Linde Molecular Sieve Type 5A) and placed under vacuum for one week.

Immediately after the desiccators were opened the bottles were capped and weighed and divided equally between seven desiccators containing sulphuric acid solutions as described in Section 4, (a), (ii). After one week the desiccators were opened and the bottles were capped and weighed. The bottles were returned to the desiccators until the products revealed constant weights, additional weighings being made every second day.

All the samples reached weights constant to less than ±.0005 g. in eleven days or less. When all the weighings were completed, the densities of the sulphuric acid solutions were measured to determine the extent to which the concentrations had changed due to the uptake of water by the samples. Moisture contents were calculated on a dry weight basis and plotted as functions of relative humidity.

(iii) Humidification and final desorption velocities. To measure humidification velocities we used the apparatus depicted in Figure 7 with the incorporation of two temperature controllers not used in the determination of the rate of freeze-drying at room temperature. The valves V_1, V_2, V_3 (Figure 7), closed after the completion of the freeze-drying velocity measurements, were left shut, the condenser C allowed to warm, and the ice inside it to melt. Partly electrical, partly pneumatic systems (Luyet and Rapatz, 1957) were then set up to control the temperatures of the condenser C and sample chamber SC (thus also the temperatures of the water and the suspended freeze-dried material, respectively). Temperatures were chosen such that, on opening the valves V_1 and V_3, and the needle-valve NV, the relative humidity tended in the sample chamber, to reach that value which caused the sample in question to demonstrate, after compression, the best recovery on rehydration. The requisite relative humidities were determined according to methods already described. See Section 4, (a). Plots showing increases in the quantities of sorbed water as a function of time were prepared in all fourteen cases — seven foods × two freezing rates × one final (best) humidity.

To measure "final drying" velocities, the foods resorbed at relative humidities best for compression were removed from the basket suspended in the specimen chamber, compressed to 500 p.s.i. and returned, in the basket, to the sample chamber.

NOTE: In some cases, food pieces containing 3 mil copper constantan thermocouples, inserted prior to freezing,

were incorporated in the 20 g. quantities placed in the wire mesh baskets just mentioned. The leads were connected to wires attached to the beam of the recording balance (not affecting the operation of the balance) and afforded records of ice interface temperature during freeze-drying, temperatures during desorption, and temperature rise on exposure of dry material to high relative humidity.

(b) Processing by limited freeze-drying.

(i) Desorption isotherms. The data from which the desorption isotherms could be constructed were obtained, as were the rates of freeze-drying at room temperature, in the apparatus shown in Figure 7. The apparatus was operated with simultaneous control of the temperatures of the sample chamber and the condenser. In a typical case, 5 to 10 g. of food were placed in a wire basket of the same design used in the previous experiments, suspended in the specimen chamber from the balance beam and warmed to -10°C. With the condenser primed with ice and adjusted to -14.6°C., valves V_1 and V_3 were opened and valve NV adjusted to permit limited freeze-drying to proceed. Adjustment of NV was made by reference to the readings of the vacuum gauges G_1 and G_2 such that: (1) air did not remain in SC, (2) evaporative cooling of ice in C by transfer to the trap T could not occur.

When, after one day or more the weight/time trace showed the total sample weight not to be decreasing any further the condenser temperature was reduced to -16.6°C. (representing 50% R.H. in the SC at -10°C.). When a further plateau was detected in the weight/time curve the condenser temperature was once more lowered. In this manner, samples were exposed in succession, to relative humidities of 60, 50, 40, 30, 20, 15, 10 and 5% such that equilibrium weights were obtained at each humidity*. Moisture contents based on the weight at 0% R.H. were plotted as functions of R.H. to produce desorption isotherms. The desorption isotherms provided a means of determining the condenser temperature such that limited freeze-drying at -10°C. yielded a sample of a given residual water content by direct desorption.

---

*We have followed the practice of defining relative humidity below 0°C. with reference to vapor pressures established by pure supercooled liquid water, not by ice at the temperature in question.

(ii) Determination of the velocities of limited freeze-drying and of final drying after compression. When the results of the compression/restoration tests on foods prepared by limited freeze-drying (see Section 4, (b), (ii)) were known; and the most appropriate condenser temperatures for each material could be selected, 20 g. quantities of these foods were subjected to limited freeze-drying in the apparatus shown in Figure 7, that is in the same apparatus used to measure velocities of freeze-drying at room temperature, etc. and to obtain, after limited freeze-drying, the desorption isotherms.

For each type of material examined, three separate experiments, each one involving a different condenser temperature, were completed. The three condenser temperatures were selected in each case to yield the three relative humidities, the upper, the mean and the lower, desorption to which produced the water contents most desirable with reference to compression/restoration behavior. The use of three separate sets of conditions for each starting material permitted the measurement of the effect of the driving force on the drying rate. As in the study of the foods humidified after freeze-drying at room temperature, each material, after compression at room temperature, was returned to the vacuum system for a determination of the final drying rate.

6. Freeze-Drying and Associated Operations — Quantity Production for Sensory Evaluation.

(a) Freeze-drying at room temperature.

(i) Freeze-drying at room temperature. Freeze-drying of 4-pound quantities of various foods at room temperature destined for the taste panel was, like the freeze-drying of the 100-g. quantities, conducted in the apparatus shown in Figure 2. It was found in general that the 2-liter flasks could, quite easily, be loaded with as much as 1 pound of frozen food each and still offer an efficient means of freeze-drying.

As in the production of the smaller quantities the freeze-drying was carried out with dry ice in the cold finger type condenser. Completion of freeze-drying was judged according to the criteria (1) that the flasks no longer feel cool to the hand, (2) that the readings from gauges G_1

and G_2 should be equal. Due to the length of the runs and the high sample loadings (from 24 to 36 hours in some cases) sorption pumping was used in place of mechanical pumping (1) to maintain the level of contaminant gases below the 1 micron level, (2) to effect a reduction of noise in the laboratory.

Completely freeze-dried materials (less than 1 g. of residual water per 100 g. dry weight) were removed and stored under argon to await transfer to desiccators for controlled remoistening.

(ii) Humidification. This was performed in vacuum desiccators, over sulphuric acid solutions at 25°C. in the manner described for small test quantities except for the following procedural differences: (1) larger desiccators (21 cm. i.d.) were used; one per food product per relative humidity, (2) sulphuric acid solutions generating precisely the required humidities were poured into the desiccators, (3) weights of water equal to those to be taken up by the foods (the values were obtained from the dry weights of the foods and the resorption isotherms) were added slowly to the sulphuric acid solutions, (4) when the foods were sealed in the desiccators the sulphuric acid was stirred slowly and continuously, (5) humidification was terminated after 16 to 20 hours (the resorption velocity measurements had shown longer periods to be unnecessary).

(iii) Compression. This was accomplished 250 ml. at a time in an aluminum cylinder having smooth-faced aluminum discs, closely fitting the cylinder walls, for sliding ends (Figure 8). The cylinder was filled as quickly as possible with the freeze-dried food having the desired moisture content, closed and inserted in the press. Materials were in all cases subjected to 500 p.s.i. of sample area for one minute, after which pressure was quickly released.

(iv) Final drying and storage. Discs of compressed material were measured for thickness, weighed, dried in vacuum desiccators at 25°C. over dry Molecular Sieve, and reweighed to permit calculation of the bulk densities. Storage was effected in vacuum, over dry Molecular Sieve, at 2°C.

(b) Limited freeze-drying.

(i) Limited freeze-drying. Several-pound quantities of each food were processed at a time in the apparatus represented in Figure 3. This apparatus was made, in part, from the one used

for freeze-drying at room temperature. The vacuum system was assembled in a constant temperature room maintained during production experiments at -10±0.5°C. The only parts of the apparatus not installed in the room at -10°C. were the pneumatic regulator PR and the multipoint recorder MR. Forced circulation of the air in the room caused the sample chambers (the 2-liter flasks) to warm to -10°C. towards the end of a run. Any desired difference between cold room and condenser temperatures could be preset and maintained with the aid of the penumatic regulator PR, the copper coils CC_1 and CC_2, and the solid carbon dioxide supply located as shown.

To make a run, the four 2-liter flasks were loaded from storage at -40°C., carried to the room at -10°C. and attached by silicone rubber o-rings to the apparatus. When the air was pumped out of the system and the valve closed, the needle valve was adjusted to yield a reading at G_1 of 25 to 50 microns (while G_2 registered a pressure in the order of 1 mm., depending on the condenser temperature employed). In this way, limited freeze-drying was conducted in a cold room in a manner permitting economic use of equipment and services. To test for completion of drying (1) the frost which accumulated on the outside surfaces of the 2-liter flasks was allowed to disperse by slow sublimation to the surroundings, (2) vacuum was broken with argon and the flasks and their contents weighed, (3) freeze-drying for 24 hours was resumed and the flasks weighed again. When a constant weight indicated a desired degree of desorption, the food was taken to a room at 25°C. to await compression.

(ii) Compression, final drying and storage. These operations were identical to those used on foods freeze-dried at room temperature.

7. Humidification of Foods Prepared by Freeze-Drying at Room Temperature — Investigation of Alternative Procedures.

While the humidification of foods freeze-dried in the conventional manner might be carried out successfully with the aid of sulphuric acid solutions, alternative procedures were clearly necessary if the process were to be "scaled up". To this end, the controlled remoistening with water vapor originating from liquid water or from ice was investigated as follows.

(a) The use of liquid water.

Depending on the temperature of the food and the required relative humidity, and providing that an equilibrium via the vapor phase could be established, there seemed to be no reason why liquid water should not be used.

Use was made of two containers, one for the food, the other for the water, thermostatted independently. The vessels, suitably connected by wide piping, could be pumped free of air by means of a vacuum system adjustable to prevent (1) loss of water vapor, (2) unwanted evaporative cooling of the source of the water vapor. Several apparatus were used to test this method.

First the freeze-drying apparatus incorporating the recording balance was used to test the practicality of the process. The temperature controllers were reset to higher-than-freezing temperatures to effect the necessary changes in the apparatus (Figure 7).

Next another freeze-drying apparatus was converted, by the addition of wide-span temperature controllers, to the same mode of operation. Dried samples were placed in the specimen chamber and the system evacuated. Equilibration was allowed to proceed at suitable sample and water temperatures for several-hour and overnight periods and the effectivenss of the rehydration evaluated by the method of drying to constant weight. The results were compared with those obtained over the sulphuric acid solutions. For the sake of these tests per se, it was not important that a freeze-drying apparatus was used to test the practicality of the process. The principal aim was to test the feasibility of the transfer of water vapor <u>in vacuo</u>.

(b) The use of ice.

Various apparatus were tested for their ability to permit the transfer of water vapor from ice to foodstuff and to provide appropriate humidities, as required, in the vicinity of a sample.

First, the same two apparatus just described in connection with the control of liquid water temperature were examined. Then, there being fewer limitations in the design of apparatus to contain ice than in apparatus to contain liquid water, other systems were investigated.

In particular, the apparatus for freeze-drying at room temperature was employed either immediately after freeze-drying or, primed with ice, to moisten foods dried in a different piece of equipment. Ice suspended on a cold-finger type condenser, or on a series of metal coils, can only be used, however, when the sample temperature-ice temperature difference is small enough to create the higher relative humidities sometimes required in humidification.

To achieve these smaller differences, and to keep the ice temperatures safely lower than 0°C. it was necessary to keep the temperature of the 2-liter flasks between +5° and 0°C. It was, in practice, found easier to keep the 2-liter flasks at 0°C. by running the entire apparatus in a room at 0°C.±.5°C. and to vary the humidity by controlling the condenser temperature at some lower value (c.f. the use of the same apparatus in a room at -10°C. for the purpose of limited freeze-drying).

To combine freeze-drying at room temperature and rehydration with water vapor from ice on the condenser the following operations were completed:

(1) The apparatus was set up in a room which could be cooled to 0°C.

(2) Freeze-drying at room temperature was conducted at a room temperature of 20°C.

(3) The temperature of the room was lowered to 0°C. when tests showed freeze-drying to be complete; the dry ice in the condenser was then replaced with ethanol precooled to -10°C.

(4) Valve V was shut and valve NV opened.

(5) Cooling coils and stirrer were placed in the alcohol and connected to a pneumatic regulator outside the room.

(6) The ice temperature was adjusted to such a value that the desired relative humidity was achieved in the four two-liter flasks.

(7) Vacuum was broken the following day with argon at 0°C.

(8) The flasks and their contents were removed to a room at 25°C.

8. Compression in the Presence of Water Vapor Only.

A special chamber was designed and constructed to test the ease with which foods might be compressed in vacuum and to determine whether or not there are any net advantages. The chamber was so designed that it could serve also as a site for freeze-drying and/or humidifying. Two modes of operation were employed.

(a) After humidification only.

Freeze-drying was completed in a separate apparatus and food was transferred at room temperature to the chamber set up as in Figure 9. Humidification was achieved with the piston clamped in the raised position by control of sample chamber and condenser temperatures. Then, when it was desired to effect compression, the valve V was closed, the elbow above it removed, and the chamber, under vacuum, placed in the press located on the same bench.

Forces of 500 p.s.i. of piston area were used for compression. The vacuum chamber itself was not subject to compression. In some cases, thermocouples were inserted in the foodstuff prior to compression and the temperature rise during compression measured in vacuum, and at a pressure of 1 atmosphere in argon.

(b) Compression in the freeze-drying chamber at the desired water content and in the original vacuum.

Procedures were devised whereby freeze-drying at room temperature, humidification, compression, and final drying could all be carried out in one vessel. (With certain alterations in these procedures, limited freeze-drying compression, and final drying were completed in the same apparatus.) In each case pilot sample quantities of about 200 g. per run were used.

The apparatus consisted of an arrangement of components already described except that the chamber employed for compression was fitted with a ring-type piston rather than a solid disc (Figure 10A). Fitting through the hole in the ring-type piston was a perforated stainless steel cylinder, specially made (1) to act as a guide for the piston movement, (2) to restrict the food to the annular space as shown in Figure 10B, (3) to serve as a conduit

for water vapor during freeze-drying, rehydration (when necessary), and final drying.

When the apparatus was operated to permit freeze-drying at room temperature, the sample chamber was held in place with a laboratory jack and the condenser was surrounded by liquid nitrogen. Humidification was achieved by the thermostatting of the condenser (C) and the sample chamber (SC) as described. Compression was effected as described in the previous section. The elbow connecting C and SC was then replaced and final drying obtained with the piston once again in the raised position, C being surrounded a second time with liquid nitrogen.

During limited freeze-drying C and SC were each maintained at preset subzero temperatures until, on completion of the process, and the closure of the valve V, the chamber and its contents were allowed to warm preparatory to compression. Final drying was effected with the condenser at -196°C. in the manner already described.

Both methods were tested repeatedly with batch quantities sufficient for taste panel evaluation.

9. Cytological Studies.

To increase understanding of the effects of compression, well-established techniques were employed to reveal the structure, under the microscope, of the compressed, and the restored materials. Dry specimens (freeze-dried or freeze-dried and compressed) were vacuum embedded in paraffin wax, m.p. 56 to 58°C. Wet samples (both the untreated controls and those treated, variously, but rehydrated) were fixed in formalin-acetic acid-ethyl alcohol (F.A.A.), dehydrated in graded mixtures of water, ethanol, and n-butanol, and infiltrated with paraffin, M.P. 56 to 58°C., at 60°C.

Longitudinal and transverse sections 10, 15 and 20 microns in thickness were cut on a rotary microtome with a steel blade. Ribbons consisting of serial sections were attached to glass slides with Haupt's adhesive and stained with saffranin O and fast green F.C.F. All the procedures were according to Jensen (1962) and Sass (1964).

Carrots, peaches, peas and shrimp were each examined after each of four or more of the following treatments:

(1) Brief storage in the fresh state.

(2) Freezing and thawing.

(3) Cooking.

(4) Cooking, freezing and thawing.

(5) Cooking, freezing, and freeze-drying

(6) Cooking, freezing, freeze-drying and humidification.

(7) Cooking, freezing, freeze-drying, humidification and compression.

(8) Cooking, freezing, freeze-drying, humidification, compression and rehydration.

Two or more of the processes (1) to (6) were chosen in each case to provide control specimens with reference to which sections prepared by methods (7) and (8) might be compared.

The slides were all examined at magnifications of 35, 100 and 500×, and photographed.

10. Studies of the Effects of Solvent Extraction.

Experiments were undertaken to determine the behavior of the various insoluble structural components of different foods, during compression, in conditions in which certain soluble components were absent. Solvent extraction was used to effect the removal of lipids, after freeze-drying, from raw beef, cooked beef and cooked shrimp and of sugars and other water soluble substances from raw cabbage, cooked carrots and raw peach. The methods were as follows.

(a) Lipid extractions.

These extractions were conducted in conventional, all glass Soxhlet-type extractors having thimble capacities of 50 ml. and 300 ml. reservoirs resting in

glass fiber-insulated electric heaters. In a first series of extractions four types of sample (beef, raw, rapidly frozen; beef, raw, slowly frozen; shrimp, cooked, rapidly frozen; shrimp cooked, slowly frozen — all freeze-dried at room temperature) were extracted for 48 hr. with 1:1 ethanol/diethyl ether mixtures, v/v. In a second series, 6 types of sample (beef, raw, rapidly frozen; beef, raw, slowly frozen; beef, cooked, slowly frozen; beef, cooked, rapidly frozen; shrimp, cooked, rapidly frozen; shrimp, cooked, slowly frozen — all freeze-dried at room temperature) were extracted with 2:1 chloroform/methanol, v/v. Generally 5 to 10 g. quantities were extracted with total solvent volumes of 200 ml.

The ethanol/ether extracted samples were used for macroscopic study under low power steromicroscopes. The chloroform/methanol extracted specimens were treated as follows.

The raw and the cooked beef, frozen, freeze-dried and solvent extracted was placed over sulphuric acid solutions in desiccators yielding, on establishment of equilibria, 20, 30, 40, 50 and 60% R.H. at 25°C. The shrimp were placed in desiccators providing 50, 60, 70 and 80% R.H. Each was left for 24 hr. to reach constant moisture content. When the desiccators were opened the samples were subjected to pressures of 500 p.s.i. and examined for their abilities to regain initial shapes, sizes and textures on immersion in distilled water. Behavior was compared with results obtained with freeze-dried foods not subjected to solvent extraction.

(b) Extraction of sugars.

Cabbage, carrots and peaches, slowly and rapidly frozen, and freeze-dried were rehydrated in distilled water and chopped into pieces having at least one dimension not exceeding 5 mm. Approximately 50 g. of each of the 6 rehydrated materials were then extracted at +2°C., the cabbage and carrots in 1-liter volumes of distilled water and the peaches with the same quantities of 0.1% aq. ascorbic acid. Extractions were conducted for total periods of 2 weeks during which times the extractants were changed three times and the systems maintained free from microbiological growth by the periodic addition of small quantities of toluene.

Finally all the samples were freeze-dried a second time and distributed among a number of desiccators containing sulphuric acid solutions. Cabbage was exposed in this way to relative humidities of 20, 30, 40, 50, 60 70 and 80% R.H., carrots to 50, 60, 70 and 80% R.H., and peaches to 20, 30, 40, 50, 60, 70 and 80% R.H. prior to compression/restoration behavior tests of the type just described for beef and shrimp.

11. Scanning Electron Microscopic Studies.

Some experiments of a somewhat exploratory nature were made to see whether or not the scanning electron microscope was well suited to the study of the effects of compression. Since samples as large as 1 cm. × 1 cm. × 1 cm. can be placed in the scanning type instrument and examined directly, without the need of thin sectioning or of replication, the following methods were employed.

(a) Control specimens.

Foods frozen both ways, i.e., slowly in air at -40°C. and rapidly by one of the three methods described earlier, were taken from storage at -40°C.,removed to steel dewar-type vessels and cooled to -196°C. Pieces were cleaved at -196°C. with the aid of a sharp, clean steel chisel, positioned according to the cross-section required and struck with such a force that the sample was not splintered. The freeze-fractured specimens produced in this way were freeze-dried at a sample chamber temperature of -40°C. until tests by the method of v.p.r.t.e. showed sublimation to be complete (see p.8).

Freeze-dried, freeze-fractured specimens were removed from the sample chamber inside a "dry box" (2 to 5% R.H.), inspected under a low power microscope, mounted with a cellulose nitrate cement on aluminum specimen holders, and coated in vacuo with a several hundred angstrom layer of gold-palladium alloy. No further preparation for viewing was needed.

(b) Compressed specimens.

In order to expose the effects of compression in a representative way a post-compression fracture was conducted as follows. Freeze-dried foods were humidified to yield suitably moist materials for best compression behavior. Compression was conducted in the manner already described, after which the freshly compressed samples were quickly cooled to -196°C. and fractured under liquid nitrogen. Fractured pieces were allowed simultaneously to warm and to lose remaining water over a desiccant (Linde Molecular Sieve) in a glass vacuum desiccator set aside at 25°C. Warm, dry, compressed and fractured samples were, like the controls, handled, inspected and cemented to the aluminum stubs in the dry box and coated in vacuo with gold-palladium.

To provide the continuous metallic coating necessary to render the sample surfaces electrically conducting under the scanning electron beam all the samples were continuously rotated, on their supports, during the deposition of the heavy metal alloy. After the coating operation, samples were stored and, when necessary, transported in glass vacuum desiccators charged with Molecular Sieve.

RESULTS

The results obtained in the course of the Phase 1 studies will be described in this section under the same 11 headings which characterized the "Methods" section. Crosscorrelations of the data presented under the different headings will be deferred to the "Discussion" and "Conclusions" sections.

1. Cooking.

The thermal processing of beef, carrots, and peas was completed entirely according to plan. Each foodstuff was judged (by an informal panel) to be properly cooked and to constitute a highly acceptable component item. Time-temperature curves obtained during the cooking of the beef are typified by the recording reproduced in Figure 11.

2. Freezing.

The slow freezing of the various foods was completed in air at -40°C. without difficulty. The cooling curves obtained in certain cases indicate the freezing rates and should permit comparisons with freezing procedures used in other laboratories.

Evolution of the latent heat of fusion gave rise to the well-known "plateau" event more in some foods than others. Figures 12, 14, 16, 18 & 20 show how the time spent near the freezing point varies with the type of sample, being about 8 minutes for raw beef, 6 minutes for cooked beef, 25 minutes for carrots, 15 minutes for peaches, and 15 minutes for shrimp.

Peaches frozen according to the first of the three "rapid" methods described, retained both natural color and form. The cooling curves obtained, shown in Figure 19, indicated a freezing rate 4 to 5 times greater than that obtained when peaches were frozen "slowly" in air at -40°C.

Peas, frozen by immersion in liquid nitrogen did not crack or split. Marked whitening was, however, observed. While cooling curves could not for practical reasons be obtained it is estimated that freezing was completed in about 1/100 of the time required in air at -40°C.

Other foods were rapidly frozen by admixture with dry ice. The method permitted a rapid freezing; at the same time it yielded materials devoid of cracks and free from fragmentation. Evidently the biological structures tolerated the mechanical stresses developed. Beef, raw and cooked, cabbage, carrots and shrimp were readily handled. Moderate color losses were observed with beef, carrots and shrimp. Cooling curves obtained with beef (Figures 13 and 15), carrots (Figure 17) and shrimp (Figure 21) indicate the cooling rates achieved. Evidently rapid freezing resulted in cooling rates 5 to 20 times greater than those obtained in slow cooling, depending on the food and the way in which the cooling rate is defined. These differences in cooling rate determine not only differences in the size of the ice crystals formed but also their location.

3. Storage.

The results of the storage at -40°C. were entirely satisfactory. Cold rooms operated without interruption

at -40±1°C. Foods were preserved unchanged for periods of up to one year. Only in a few instances was the migration of ice from food to polyethylene surface apparent. Neither freezer burn nor collapse (viscous flow of solute) was observed on the surface of any food.

4. Freeze-Drying and Associated Operations — Physical Compression/Restoration Studies.

(a) Behavior of small test quantities upon freeze-drying at room temperature and during humidification.

Freeze-drying, in an apparatus set up to resemble commercial equipment, produced, in each case (7 foods, 2 freezing rates each), material of a totally satisfactory nature. In no case (not even with peaches) was there any indication of structural collapse, melting, or distortion due to drying. Neither did the various materials crack or fragment.

Humidification (each of the 14 products being exposed to each of 7 relative humidities) resulted in different behavior, depending on the food and the humidity. Cabbage exposed to 60% R.H., or higher, underwent a spontaneous shrinkage; carrots, at 50% R.H., or higher, were rapidly bleached. Peaches spontaneously collapsed at 50% R.H., or higher. Several foods developed most objectionable off-flavors on equilibration at higher humidities.

(b) Behavior of small sample quantities upon limited freeze-drying.

Raw beef, cooked beef, peas and shrimp exhibited excellent behavior upon limited freeze-drying when conducted at -10°C. The freeze-dried product resembled the frozen material in size, shape and color regardless of the relative humidity to which the limited freeze-drying was taken. Carrots demonstrated a slight shrinkage and a deepened color, neither of which was rated unacceptable. Cabbage and peaches upon limited freeze-drying at -10°C. yielded drastically shrunken, distorted material with much evidence of internal structural collapse. Efforts to

subject these products to limited freeze-drying at lower temperatures were equally unsuccessful. Neither cabbage nor peaches could be dried at a sample chamber temperature of -20° or -30°C. (with an appropriately lower condenser temperature in each case) without the formation of a grossly collapsed freeze-dried product.

(c) Compression/restoration behavior.

These preliminary results are summarized in Tables I and II. Behavior is listed with reference to food, to method of preparation, and to relative humidity. Since freezing-rate, within the limits between which we varied it, had no measurable results on compression/ restoration behavior, separate columns for slowly and rapidly frozen materials have been omitted.

Perfect restoration was not observed either in raw or cooked beef at any humidity. Moderate to good recovery was, however, observed over a rather wide range of humidities both after freeze-drying at room temperature and after limited freeze-drying. The choice of relative humidity was not as critical as in some other foods.

In cabbage satisfactory behavior was attained only at one relative humidity after freeze-drying at room temperature.

Excellent behavior was observed in cooked carrots to extend across rather broad ranges of relative humidity.

Nothing better than very slow recovery could be obtained with peaches.

Peas restored very well but behavior on compression was more sensitive to relative humidity than it was, for example, with carrots.

Best restoration to normally textured shrimp appeared to be critically dependent on relative humidity. Restoration was, however, observed to take place after compression at sub-optimal humidities and to yield remarkably tender material.

TABLE I Effects of Water on Freeze-Dried Foods Compressed after Remoistening to Various Relative Humidities

R. H. (%)*	beef (raw)	beef (cooked)	shrimp
80	failed to restore	failed to restore	no restoration
70	near total failure to restore	near total failure to restore	no restoration
60	partial failure to restore	partial failure to restore	fair restoration
50	good recovery	good recovery	good restoration
40	good recovery	good recovery	good restoration
30	partial recovery	partial recovery	fair restoration
20	crumbled on compression	crumbled on compression	partially fragmented; partially restored

* Freeze-dried materials were exposed to atmospheres of these humidities at 25°C. prior to compression at 25°C.

TABLE I (continued) Effects of Water on Freeze-Dried Foods Compressed after Remoistening to Various Relative Humidities.

R. H. (%)*	cabbage	carrot	peach	peas
80	failed to restore	poor recovery	no restoration	poor recovery
70	near total failure to restore	poor recovery	no restoration	no damage; excellent recovery
60	failed to restore	excellent recovery	no restoration	no crumbling; good restoration
50	little response to water	good recovery	no restoration	some crumbling; some restoration
40	restored but to rubbery texture	some cracks; partial recovery	very poor restoration	endocarp crumbles
30	wets and restores good texture	cracked little tendency to restore	no crumbling; poor restoration	endocarp crumbles; no restoration
20	wetted with difficulty	pieces cracked; failed to restore	finely fragmented; no restoration	endocarp fragments; no restoration

* Freeze-dried materials were exposed to atmospheres of these humidities at 25°C. prior to compression at 25°C.

TABLE II. Effects of Water on Foods Compressed after "Limited Freeze-Drying" to Various Relative Humidities.

R. H. (%)*	beef (raw)	beef (cooked)	carrots	peas	shrimp
60	no recovery	no recovery	partial recovery	poor recovery	no recovery
50	partial recovery	partial recovery	good recovery; fair texture	good recovery; poor texture	no recovery
40	partial recovery	partial recovery	excellent recovery; excellent texture	good recovery good texture	poor recovery
30	partial recovery	partial recovery	excellent recovery good texture	split; poor texture	fair recovery
25	partial recovery	partial recovery	fair recovery	fragmented; fail to recovery	fair recovery**
10	best recovery	best recovery	fragment on compression	powder upon compression	good recovery***

*Relative humidities were measured at -10°C. Compression was conducted at 25°C.

**Slowly frozen shrimp more nearly recovered than did rapidly frozen shrimp.

***Rapidly frozen shrimp exhibited better restoration after compression at this humidity than did slowly frozen shrimp.

V. Freeze-Drying and Associated Operations — Quantitative Data

(a) Resorption isotherms.

The data are presented in the form of 7 curves denoting the binding of water on exposure of freeze-dried materials to atmospheres of precisely controlled relative humidities. The plots are seen in Figures 22 to 28 and are indicated in each case by 'R'.

The freezing rate was found not to affect the isotherms. Points fell so closely together in every case that separate curves, one for each freezing rate, could not be drawn.

(b) Desorption isotherms.

These are shown in Figures 22, 23, 25, 27 & 28 and are indicated in each case by 'D'. The data represent the extents to which the freeze-dried materials continue to bind water upon limited freeze-drying to various relative humidities indicated. Here also there is an absence of any effect of freezing rate on the isotherms.

The consistent and very considerable relative displacement of the two types of isotherm is shown in each of five of the seven figures. In the case of the carrots and the peas, the similarity between the corresponding pairs of isotherms is evident. The close correspondence of the respective isotherms in the case of the raw beef, the cooked beef and the shrimp is also seen. In the case of the beef, the vertical separation of the curves D and R, increases as a result of cooking.

(c) Velocity measurements.

(i) Freeze-drying at room temperature. These determinations on raw beef, cooked beef, cabbage, carrots, peaches, peas and shrimp are reproduced in graphical form in Figures 29-37. The data points represent places at which measurements were taken from the original recordings; the original velocity measurements were continuous. The times taken by the samples to freeze-dry to 1/2 their original weight, and the times taken to lose 90% of the ice first formed,

were calculated. These values were used to provide a frame of reference for the data derived from measured rates of limited freeze-drying.

(ii) Limited freeze-drying. The determinations of the velocities at which raw beef, cooked beef, carrots and peas underwent limited freeze drying are shown in Figures 29, 30, 32, 33, 35 and 36. They may be compared directly with the velocities with which the same materials freeze-dry at room temperature. Times to dry to half the original weight and times to loss of 90% of the ice originally present were calculated from these data and may be found in Tables III and IV.

(iii) Humidification velocities. The times taken by fully freeze-dried materials to resorb water from the vapor phase were determined from weight/time recordings. Times taken to regain 9/10 of the weight predicted from the resorption isotherms, upon exposure to sources of controlled relative humidity, were in the range 0.5 to 6 hr., being the least for cooked and raw meat, and shrimp (0.5 to 1 hr.), higher for vegetables (ca.2 hr. for carrots, see Figure 39) and highest for peach (6 hr., see Figure 38).

(iv) Final drying velocities. As in humidification the muscle tissues underwent more rapid changes in weight with change in humidity than did the other foods, being essentially dry one to two hours after compression.

Carrots and peas dried much more slowly after compression. Figures 39 and 40 illustrate the effect of compression on the rate of the desorption drying of carrots and peas, respectively. Note that, under high vacuum at 25°C., both compressed products are still losing weight after fifteen hours. Carrots not compressed were dried after 6 hours, peas after 15 hours.

6. Comparison of Effects of Different Processing Methods and Conditions on Organoleptic Properties.

(a) Carrots.

Quantities sufficient for taste panel evaluation were processed according to the methods described in the previous

TABLE III. Times to Freeze-Dry to Half the Original Weights, Expressed as Multiples of Times Taken when Freeze-Drying was Conducted at Room Temperature to a Final Relative Humidity of 0%.

Food Product		Final Relative Humidity in the Sample Chamber				
Food	Freezing Rate	0%	10%	30%	40%	50%
Beef, raw	slow	1	2.8	-	-	-
Beef, raw	rapid	1	2.6	-	-	-
Beef, cooked	slow	1	3.2	-	-	-
Beef, cooked	rapid	1	2.5	-	-	-
Carrots	slow	1	-	5	-	8
Carrots	rapid	1	-	5	6	8
Peas	slow	1	-	-	5	-
Peas	rapid	1	-	3	5	6

TABLE IV. Times for 90% of the Ice Originally Present to Sublime, Expressed as Multiples of Times Taken when Freeze-Drying was Conducted at Room Temperature to a Final R.H. of 0%.

Food Product		Final Relative Humidity in the Sample Chamber				
Food	Freezing Rate	0%	10%	30%	40%	50%
Beef, raw	slow	1	2.7	-	-	-
Beef, raw	rapid	1	2.6	-	-	-
Beef, cooked	slow	1	2.9	-	-	-
Beef, cooked	rapid	1	2.6	-	-	-
Carrots	slow	1	-	4.5	-	8
Carrots	rapid	1	-	4	6	8
Peas	slow	1	-	-	5	-
Peas	rapid	1	-	4	5	6

section to yield discs 7.5 cm. in diameter, ca. 1 cm. thick. Bulk densities of about 0.8 were obtained in each case. Compressions were conducted at moisture contents (1) less than, (2) equal to, (3) higher than those found in preliminary tests to yield best restoration. The 12-man taste panel rated the differently processed carrots, rehydrated for 5 minutes, as shown in Table V. The panel rated the foods according to the following hedonic like-dislike scale:

7 - Like Very Much

6 - Like Moderately

5 - Like slightly

4 - Neither Like Nor Dislike

3 - Dislike Slightly

2 - Dislike Moderately

1 - Dislike Very much

Table V

Mean Scores:

Dried uncompressed control: 2.9

Desorbed to 30% R.H.,-10°C: 2.8

Desorbed to 40% R.H.,-10°C: 3.0

Desorbed to 50% R.H.,-10°C: 3.9

Resorbed to 50% R.H., 25°C: 2.7

Resorbed to 60% R.H., 25°C: 3.0

Resorbed to 70% R.H., 25°C: 3.4

Significance:

Differences are significant (P=0.05)

Least significant difference: 0.8 (P=0.05)

Clearly the material compressed at the highest moisture content was preferred in each series. There is also, at

the highest moisture content, an indication that the carrots compressed after limited freeze-drying were preferred over those compressed after freeze-drying at room temperature and humidification.

(b) Peas.

7.5 cm. diameter discs, approximately one cm. in thickness, and having bulk densities in the range 0.75 to 0.8 were prepared by freeze-drying at room temperature or by limited freeze-drying.

The results (Table VI) permit comparison of (1) the behavior of samples compressed at different moisture contents (2) the behavior of samples prepared for compression by conventional and by limited freeze-drying. The peas were allowed to rehydrate for 10 minutes.

Table VI

Session I

Mean Scores:

Control, dried, uncompressed:	3.6
Resorbed to 60% R.H., 25°C:	3.8
Resorbed to 70% R.H., 25°C:	3.9
Resorbed to 80% R.H., 25°C:	4.4

Significance:

Differences are not significant.

Session II

Mean scores:

Control, dried, uncompressed:	2.2
Desorbed to 30% R.H.,-10°C:	4.8
Desorbed to 40% R.H.,-10°C:	4.5
Desorbed to 50% R.H.,-10°C:	4.7
"Desorbed-Resorbed" :	3.8

Significance:

Differences are highly significant (P-0.01).

Least significant difference 1.0 (P=0.05).

It appears that peas freeze-dried at room temperature restore best when exposed to 80% relative humidity prior to compression. Limited freeze-drying seems, on the other hand, to yield material that restored equally well after compression at each of 3 humidities.

Like carrots, peas compressed after limited freeze-drying appeared to restore better than those compressed following a freeze-drying at room temperature.

Neither cabbage nor peaches could be prepared by limited freeze-drying except at a very low sample chamber temperature. Comparisons of the effects of different processing methods on the behavior of these foods could not therefore be completed.

One series of experiments was conducted to determine the effect of temperature of compression on the moisture content best for compression. Slowly frozen, cooked beef, freeze-dried at room temperature was divided between desiccators containing aqueous sulphuric acid some of which were maintained at 25°C., some at 2°C. Best recovery of the pieces equilibrated and compressed at 25°C. was obtained in the range 30 to 60% R.H. with most acceptable results at 40% and 50% R.H. Good results were obtained with samples compressed at +2°C. when they were first equilibrated at relative humidities in the range 60 to 80% with best results at 60 and 70% R.H. Compression at a lower temperature must, it appears, be conducted at a higher moisture content to insure best recovery.

To test further the effect of temperature on compression and quality following restoration, quantities of beef sufficient for the taste panel were freeze-dried at room temperature, and exposed either to 70% R.H. at +2°C. or to 50% R.H. at 25°C., each humidified material being compressed at the temperature at which equilibration took place. Each material, with the necessary control samples, was submitted, after final drying, to the taste panel. Rehydration was allowed to proceed for 25 minutes before the samples were served to the panel. The panel's evaluation is shown in Table VII.

Table VII

Mean Scores:

Frozen Control: 3.9

Freeze-dried control: 4.5

Compressed at 70% R.H., 2°C: 4.3

Compressed at 50% R.H., 25°C: 4.1

Significance:

Differences are not significant.

7. Humidification in the Freeze-Drying Chamber.

(a) The use of liquid water.

In the first of 2 runs involving the least readily remoistened material, freeze-dried peach slices equal to a pound of frozen peaches were placed in the sample chamber. When the freeze-dried material was cooled to 20°C. and the liquid water to 2°C., the water reservoir and the sample chamber were connected and the system was evacuated.

The freeze-dried peach slices, exposed thus in vacuo to a 30% R.H. were removed from the system after 6 hours and compressed to 500 p.s.i. A tendency to crumble during compression was noted, indicating an incomplete absorption of water. In the second experiment freeze-dried peach slices were maintained in circumstances similar to those just described but for an overnight period. An excellent compression was then achieved, yielding firm, dense and cohesive material.

(b) The use of ice.

Peaches, peas and shrimp were each freeze-dried to better than 1% residual moisture and rehydrated by controlled return of water vapor from the condenser to the specimens as described in the "Methods" section. Peas and and shrimp were submitted to the taste panel.

(i) Peaches. Approximately 12-oz. samples of frozen peaches were placed in the 4 two-liter flasks (Figure 3) and freeze-dried at high vacuum. The room temperature was maintained at 0°C., the condenser at -78°C. When drying was completed the condenser temperature was raised to -14°C. to yield 30% R.H. in the flasks, and maintained at that value overnight. When the humidified samples were removed from the apparatus and tested at 25°C. for ease of compression a ready compressibility was observed, indicating the required return of water to the peaches had been completed.

(ii) Peas. Approximately 400 g. of frozen peas were dried in exactly the same way except that the room temperature was maintained at -10°C. At the completion of the freeze-drying, the condenser temperature was raised from -78° to -14.6°C. to establish a 60% R.H. over the peas at -10°C. Equilibrated material, taken the following morning, compressed and dried, was submitted to the taste panel and was judged together with other peas (Table VI). The panel rated this sample closer to those resorbed at room temperature than to those prepared by limited freeze-drying.

(iii) Shrimp. A direct comparison was made of shrimp compressed after resorption over sulphuric acid with shrimp compressed after resorption with water from condenser ice. The shrimp resorbed over sulphuric acid were freeze-dried originally at 25°C.; the other sample was freeze-dried at 0°C. In both cases the condenser was maintained at -78°C. until drying was complete. The shrimp dried at 25°C. were resorbed over acid made up to yield 50% R.H. at 25°C. The shrimp dried at 0°C. were exposed to ice at -8.2°C.; that is, to a 50% R.H. at 0°C. Upon completion of 6-hr. equilibration periods, all the samples were compressed at 25°C. and subjected to final drying. The assessments by the taste panel, after a 30 minute rehydration, are shown in Table VIII.

Table VIII

Mean Score:	
Frozen control:	4.9
Slowly frozen (H_2SO_4):	4.5
Rapidly frozen (H_2SO_4):	3.8
Slowly frozen ("Desorbed-Resorbed"):	4.2
Rapidly frozen ("Desorbed-Resorbed"):	3.2

Table VIII (Continued)

Significance:

Differences are significant (P=0.05)

Least significant difference: 1.1 (P=0.05)

The results indicate the equal acceptability of the two methods of humidification. The superiority of the slowly over the rapidly frozen shrimp, regardless of the choice of further treatment is clearly evident.

8. Compression in Vacuum (and Measurement of Temperature Rise During Compression)

(a) After humidification only.

(i) Beef. Cooked beef was humidified to 30, 40, 50, 60 and 70% R.H. at 25°C. in separate experiments. Six to ten cubes, one of which contained a 3-ml. thermocouple, were employed per experiment. The temperature registered by the thermocouple was recorded in each case during compression to 500 p.s.i. in vacuo. A second series of experiments was conducted in one atmosphere of argon.

The results showed (1) that these samples underwent compression just as readily in the presence of one atmosphere of argon as they did in vacuum, (2) that the core temperatures of the cubes rose between 1.5 and 2.0 degrees C. upon compression in argon and from 0.5 to 1.0 degrees C. in vacuum. Clearly the adiabatic compression of the meat did not result in appreciable warming in either case.

(ii) Peaches. Peach slices equivalent to one pound of frozen peaches were humidified in 30% R.H. at 25°C. and compressed in vacuo. The slices were readily formed into a highly cohesive disc having a bulk density after final drying (in the same chamber) of 0.81. No difficulties of any sort were encountered in conducting the sequence, humidification, compression, final drying, in one chamber.

(b) Compression after freeze-drying.

Three 200-g. batches of frozen peas were subjected to limited freeze-drying at a 40% R.H. at -10°C. (condenser maintained at -19°C. throughout the process). When drying was completed the products were warmed to 25°C. and compressed in situ (1) at 500 p.s.i. for 1 minute under one atmosphere of argon, (2) at 500 p.s.i. for one minute in vacuo, (3) at 500 p.s.i. for one hour in vacuo. Bulk densities, after final drying, were determined to be 0.45, 0.64, and 0.72, respectively.

Evidently (1) the absence of a noncondensible gas aids the process of compression considerably, (2) a prolonged compression is more effective than one of short duration. No problems were encountered in the operation of the apparatus.

9. Cytological Investigations

(a) Carrots (Photos. 1 to 8).

Cooking appeared to cause a separation of the cells in the parenchyma, one from another, with the formation of nearly straight rifts 5 to 30 cells in length; no other changes were detected.

It also became apparent that cells were distorted and disrupted more by slow freezing than by rapid freezing, and more by compression upon equilibration at 70% R.H. than at 55% R.H. Damage took the form, variously, and without a particular pattern of incident, of (1) failure of small groups of cells to restore following compression, (2) the separation of some cells from others, along intercellular surfaces, (3) the failure of groups of cells to regain their original orientation with respect to other cells.

Thus the damage was not restricted to a single form. In the case of the more slowly frozen materials the visible damage to the cell structures correlates with taste panel assessment of the effect of moisture content prior to compression. Only cells in the xylem seem not to be affected by the different treatments.

Freeze-dried specimens embedded directly in wax did not provide any additional useful information.

(b) Peaches (Photos. 9 to 16).

Freezing and thawing caused cell walls to break and some cells to shrink considerably. Rapid freezing caused less change in structure than did slow freezing. Cell contents, in general, were caused to shrink and to persist in the form of collapsed material localized to one part of the cell. Generally the cell damage was greatest in the endocarp save for the vascular bundles which appeared to be well preserved, and least in the exocarp, where the average cell diameter was smallest.

Freeze-drying, followed by gentle room temperature rehydration, appeared to cause little more alteration in the structure than did freezing and thawing. Cell walls were seen to be ruptured here and there and frequently to be slightly wrinkled where slow freezing was employed prior to freeze-drying. Apparently the freeze-drying imparts a "set", unaltered by rehydration, to the distortion introduced during freezing.

Specimens subjected to compression showed, after restoration, little further damage where compression was used to reduce the volume of the freeze-dried material by 50%, by 75%, and by 85%. Slightly increased damage was observed with increased compression. Greater wrinkling of cell walls was found in tissues frozen slowly prior to freeze-drying. Generally, however, visible evidence of damage correlating with a loss of acceptable texture, was not obtained.

(c) Peas (Photos. 17 to 28).

By comparison with the raw control, cooked peas demonstrated detached seed coats, collapsed parenchymal cells and destruction of the cytoplasmic constituents. The appearance of the cell walls in the endocarp was generally not altered by cooking.

Studies involving the freezing and freeze-drying of cooked material, showed, by reference to "controls" cooked but not frozen, that slow freezing was much more damaging

than rapid freezing, that while rapid freezing caused only the collapse of the cells in the endocarp, slow freezing caused the same cells to become detached one from another, to break open and to lose and scatter the starch grains. Note that the sections obtained from frozen and freeze-dried material were cut from freeze-dried peas subjected to direct vacuum-infiltration with melted paraffin.

Compression, studied after restoration, caused additional damage to the cells in the endocarp, regardless of the freezing rate prior to freeze-drying. But the structures were in general better preserved when peas were rapidly frozen prior to compression and restoration.

Peas compressed at 70% R.H. yielded the best preserved structures. Those compressed at 60% R.H. crumbled so badly that, on restoration, material could not be retained in wax for sectioning. Peas compressed at 80% R.H. sectioned quite easily but demonstrated somewhat poorer retention of structure than those treated at 70% R.H. Thus, to a certain extent, the cytological results paralleled the judgment of the taste panel.

(d) Shrimp (Photos. 29 to 36).

Controls which were frozen, freeze-dried, rehydrated and fixed were hardly different in appearance from fixed, unfrozen specimens. Only in the specimens frozen rapidly prior to freeze-drying was there a partial rearrangement of muscle fibers.

Slightly more frequent evidence of a reorientation of fibers, one with respect to another was observed in freeze-dried compressed material restored in water prior to fixation. In rapidly frozen specimens, moreover, a tendency to remain compressed was noted. Furthermore, very faint fracture lines were observed in some instances to cross the fibers where the latter were seen in longitudinal aspect. Actual segmentation of the fibers was not, however, visible. Apparently the compression had damaged the fibers but had not prevented them from recoverying their original size, shape and relative location.

Slides prepared from shrimp freeze-dried and embedded directly in wax provided completely different results. Fibers were everywhere severely fractured and fragmented whether

the samples were previously compressed or not. In the circumstances the results could only be attributed to the very damaging effects of the process by which freeze-dried specimens were directly infiltrated, under vacuum, with melted paraffin.

10. Solvent Extraction.

(a) Beef, raw.

Slowly frozen, freeze-dried, solvent-extracted raw beef restored best when compressed at 30 and 40% R.H. Rapidly frozen material restored best at 30% R.H. Thus the behavior was hardly different from that of the non-solvent extracted samples.

(b) Beef, cooked.

Slowly frozen cooked beef restored about equally well regardless of the relative humidity before and during compression. Rapidly frozen solvent-extracted material restored best when compressed at 50, 60, and 70% R.H., least well when compressed at 20, 30, and 40% R.H.

Apparently the extraction of the lipids exerts a much more marked effect on the behavior of the cooked beef. It seems also that the freezing rate prior to freeze-drying determines the nature and magnitude of the effect of solvent extraction.

(c) Cabbage.

Both slowly and rapidly frozen, freeze-dried, water-extracted cabbage restored best after compression at 30% and 40% R.H., as did the unextracted materials. The behavior was, however, observed to depend on freezing rate. While the slowly frozen cabbage restored, at best, in a "poor to fair" manner, and showed also some evidence of "poor to fair" recovery after compression at 70% and 80% R.H., the rapidly frozen samples demonstrated "fair to good"

recovery at 30% and 40% and "fair" or "poor to fair" recovery at all other relative humidities.

(d) Carrots.

Slowly frozen water-extracted carrots restored best after compression at relative humidities in the range 20% to 50%, without particular distinction. Rapidly frozen material recovered best only at 30% and 40% R.H. These performances may be compared with those from which water-solubles were not extracted, the latter restoring best when compressed after exposure to 50 and 60% R.H.

(e) Peaches.

Regardless of freezing rate, peaches extracted with distilled water recovered after compression according to a complex scheme. Thus, samples compressed after exposure to relative humidities of 30 and 80% restored noticeably better than any other. Samples compressed at 20%, 40%, 50%, 60% and 70% R.H. recovered much less well. The good behavior related to an exposure of 30% R.H. correlates with the behavior of peaches not subjected to water extraction. No such correlation exists for the good behavior related to the exposure to the much higher relative humidity of 80%.

(f) Shrimp.

Slowly frozen, solvent-extracted shrimp restored best following compression upon equilibration at 70% R.H.; rapidly frozen shrimp recovered best after exposure to 80% R.H. 50% and 60% R.H., most effective in promoting recovery where solvent extraction was <u>not</u> employed, were both markedly less effective in preconditioning the extracted material.

11. Scanning Electron Microscopy.

In the series of experiments to be described, materials compressed at moisture contents best for subsequent restoration were compared in three-dimensional aspect, at magnifications up to 10,000× and a depth of focus exceeding 100 microns, with samples freeze-dried but not compressed. External and internal surfaces (the latter produced by fracture techniques) were examined. The results are described with reference to photographs (see Photos. 37 - 48).

(a) Cabbage.

The freezing in cold air at -40°C. (with freeze-drying at -30°C.) produced a tissue structure within which the arrangement of the cells was not apparently distorted. Intercellular spaces were, however, visible and there was little evidence of any dry matrix in any of the cells.

(Compressed material was not examined.)

(b) Carrots. (Photos. 37 and 38).

Uncompressed material did not appear to be damaged either by rapid or by slow freezing and consisted mostly of empty cells (after freeze-drying at -30°C.) devoid of obvious interconnections. Intercellular voids were detected in a few places. Clues concerning possible means of rehydrating freeze-dried carrots were not obtained from the study.

Compressed freeze-dried carrots demonstrated very dense packing of cell wall structures and of scattered small sub-cellular bodies. Presumably the cell walls and the material cementing these walls together contained a considerable accumulation of sugars. In any case the voids were so few as to suggest bulk densities exceeding 1.0 (confirmed by the observation that compressed freeze-dried carrots, prior to final drying, sank instantly in water).

Cell walls were seen to bend in some cases to accommodate the relative displacements resulting from compression. As in the case of the uncompressed material, the mode of rehydration (found in practice to be very rapid) was not apparent from the first examination in the scanning microscope.

(c) Peaches (Photos. 39 and 40).

Frozen in air at -40°C., peaches yielded a regular "honeycomb" structure composed of smooth, rather rounded cells flattened at points of contact, the well-defined intercellular spaces forming an apparently continuous labyrinth. Seemingly, ample space and a sufficient number of connections were present to permit rapid rehydration.

(d) Peas (Photos. 41 to 48)

Frozen in dry ice at -78°C., cooked peas freeze-dried at -30°C. showed a regular distribution of small intercellular spaces, widespread evidence of the prior presence of intercellular ice. The location of the starch grains did not appear to have been disturbed by freezing. Frozen in air at -40°C., the same food gave much evidence that extracellular dendritic freezing had caused a compression of a majority of the cells into groups of as many as ten or more in such a way that intracellular freezing was prevented. Shrinkage and distortion of individual cells were generally evident, as was a separation of some cells from each other (without, it appeared, a rupture in either cell) resulting from the growth of the ice phase.

Compression of the more slowly frozen material was found to proceed with additional distortion of the majority of the cells within the cotyledons. Palisade cells in the seed coat were, however, completely unaffected by pressures of several hundred p.s.i. Evidence was also obtained indicating extensive and continuous empty space within the compressed slowly frozen peas (bulk densities from 0.8 to 0.9). These spaces were revealed in great detail by the use of stereoscopic photographic methods.

DISCUSSION

In this section the results reported in the previous section will be analyzed with reference (1) to their validity (the value of the methods employed) and (2) to their significance. The behavior of the food during the various processes in the order in which the latter were employed in practice will be discussed along with the evaluation of the results of the various additional supporting experiments.

1. Behavior toward Conventional Freeze-Drying and to "Humidification". (Isotherms, Rates of Water Loss, Uptake and Appearances)

Conventional freeze-drying, conducted in conditions of low resistance to vapor transfer and moderate resistance to heat transfer, probably took place with more sustained evaporative cooling and yielded materials of above average quality; that is, the "collapse" and the color change associated sometimes with commercially prepared material were not observed. But the method served in any case to provide good reference foodstuffs against which the results of additional processing could be judged.

Humidification, carried out in small batches, resulted in very little condensative warming of the product. Temperature differences within the food were therefore minimal. Since sources of water vapor of constant activity were assured, the extent of the humidification must always have been the same throughout the sample, and reproducible; almost certainly there were no alterations in the isotherms due to the heat generated during sorption. In other words, the results we obtained were not the marked functions of sample size they might have been where much larger apparatus and much larger sample loadings were employed.

The value of the results, then, is in their representation of the maximum water binding capacities and the best appearances of the materials. The determination of the rates of freeze-drying at room temperature afforded a direct comparison with rates of limited freeze-drying since the determination of velocities by the latter process was conducted in the same apparatus. This matter will be discussed further in later sections.

The resorption isotherms were not found to depend on freezing rate within the range of freezing rates employed. While this result might seem to conflict with some earlier observations (Luyet and MacKenzie 1967; MacKenzie and Luyet, 1967), it is significant that, in the earlier study, the measurements were conducted on mg. quantities and the isotherms obtained in several hours. In the course of the present work, resorption experiments were completed over the course of several days, during which time, relaxation processes could have allowed the elimination of the differences ascribable to the "locked-in strains".

The rate determinations did not permit an evaluation of the effect of freezing rate on freeze-drying rate since the measurements were conducted in conditions in which the effect was minimized, a conclusion borne out by the observations.

2. Behavior toward "Limited Freeze-Drying"

The use of two temperature controllers insures the restriction of the sample temperature, throughout freeze-drying, to a value somewhere between two well-defined values usually no more than a few degrees apart. This also insures there will be no changes in the sample due to combined effects of intermediate water content and high temperature. But it introduces a new factor. The extra time of exposure to processing conditions now makes possible changes at low temperatures and intermediate moisture contents where these were not previously observed. This possibility should be kept in mind.

The quality of the curves drawn through the data points indicates, as it did in the resorption experiments, the accuracy with which desorption isotherms can be determined. It is, furthermore, not difficult to set up equipment to determine such plots. A consideration of the dependence of the vapor pressure of ice on temperature indicates, however, the need to control both sample chamber and condenser temperatures at every step, during the determination of such an isotherm, to $\pm$ 0.1°C.

The desorption isotherms are independent of the initial freezing treatment. This demonstrates, as did the resorption isotherms, that the water binding properties are a function of the nature of the materials and not of the structures impressed on the latter as a result of ice formation (at least in the range of freezing rates employed).

The rate determinations were conducted in conditions in which heat transfer to the specimens was not facilitated by contact with massive heat-conducting surfaces as it would have been in a shelf-type dryer. The runs do, however, permit an illuminating comparison of the rates at different sample chamber/condenser temperature differences, that is, at different driving forces. Furthermore, all the rates

obtained this way can be compared with the rates observed when freeze-drying was carried out in conditions of the highest possible vacuum, since, with the exception of the temperature controlling devices, the apparatus used in each series of experiments was identical (Figs. 6 & 7).

The dependence of the freeze-drying rate on the driving force could be subjected to a more precise analysis in terms of the dependence of the permeability of the freeze-dried shell on the relative humidity except that the ice core temperature of the particles was not measured. Without doubt, the relative humidity-profile within the freeze-drying food — and this profile must depend on the relative humidity of the sample surface — determines the ease with which water vapor moves through biologically compartmented structures. The conditions of freeze-drying determine not only the driving forces across the dried portion of the sample; they also determine the resistances.

Similarly, the rates of resorption and final drying, after compression, will also be determined in part by the permeability which will, in turn, be determined to some extent by the driving forces. But the driving forces during humidification were determined by the moisture contents required for compression and were therefore predetermined. Likewise, the driving forces during final drying were predetermined since they represented the differences between the water vapor pressures demonstrated by the foods, after compression, and that of ice at -196°C., the latter being very close indeed to zero.

The determinations of the rates of resorption and of final drying serve nonetheless to demonstrate the rapid movement of water into and out of freeze-dried materials. The results are certainly of practical significance.

3. The Behavior of Foods, Variously Brought to Different Moisture Contents, on Compression.

It is important, first, to discuss the validity of the small scale compression tests conducted during the course of the work, But there would seem to be little reason to suspect the results were not representative of those obtained on a much larger scale. The press, which handled

batches of approximately 250 cc. (loose-packed volume), was of standard design and construction. The 3-inch diameter plates between which the pieces of food were compressed were easy to center and thus to maintain parallel during operation. The cylinder within which the plates moved prevented the sideways extrusion of the food. To summarize, the apparatus permitted the duplication of the performance of a small, typical portion of a much larger sample.

Since samples were found very frequently to yield to compression in a discontinuous manner with gradually increased pressure up to about 300 p.s.i. but not to change significantly thereafter, 500 p.s.i. was selected as a standard maximum pressure. It is evident, however, that the response to different pressures, applied upon equilibration to different relative humidities, constitutes a most important subject. The results of such an investigation would be very valuable.

Since the freezing rate prior to freeze-drying was not found to affect the behavior of the sample upon compression, it would seem safe to conclude that, during compression, one is dealing with the properties of the materials surrounding the cavities left by the sublimation of the ice. The arrangement of the cavities appears to be of little consequence.

Equally important, one observes the dependence of the behavior upon compression, on the activity of the water in the sample, to be of much the same form, whether the samples are brought to predetermined water activities by resorption from a too-dry state or by direct desorption.

Such conclusions should encourage the application of information obtained on compression in one laboratory to the processing of foods frozen and freeze-dried in another laboratory.

Insufficient resorption after a conventional freeze-drying to near-dryness (likewise, too extensive a desorption by limited freeze-drying) yielded foods that fragmented on compression. But the fact of the sorption isotherm,

that is, the proven existence of relationships describing water contents as functions of water activity, permits a discussion of the behavior upon compression with reference to relative humidity established over freeze-dried materials.

Possibly the greatest significance of the present study is in the demonstration that behavior upon compression can be determined and classified primarily with reference to thermodynamic water activity, secondarily with respect to the water content of any given food. Thus, the most suitable conditions for compression can be determined without any measurement of water contents. The best compression could then be obtained by (1) pilot runs, (2) production runs, each involving the control only of the relative humidity.

There has been some discussion concerning a possible adiabatic heating of samples during compression with results that are deleterious to the sample quality. But the measurements of sample temperatures during laboratory-scale compression showed only a 1 to 2 degree C. rise on compression to 500 p.s.i. Since it would seem that heat transfer during compression most likely proceeds in a similar manner in commercial-scale operations, behavior during compression will not be determined by the effects of heat on the moist matrix. Neither should the final product demonstrate the effects of heat.

4. Behavior of Compressed foods upon Restoration.

The preparation and restoration of the 4-lb. and 2-lb. quantities offered to the taste panel were conducted under conditions essentially similar with respect to heat and mass transfer to those that pertain to large scale operation. The laboratory experiments with smaller quantities (100 g., more or less) involved only processing and handling in conditions common to the handling of batches of every size. The use of quantities of water greatly in excess of those required per unit weight of freeze-dried material was justified on the grounds that soluble components were probably not extracted during the early stages of rehydration and restoration. Probably the greater dilution of the "solubles" in the smaller scale rehydration studies did not

influence appreciably the course of the restoration. We conclude that the small scale experiments provided results representative also of the behavior of larger quantities.

In every instance, there did exist a relative humidity exposure to which, prior to compression, allowed a best restoration. Markedly poorer restoration could be detected when foods were prepared by exposure to relative humidities outside an interval comprising 10 to 20 percentage points. The size of the percentage interval within which preparations yielded good restoration was observed, moreover, to be smaller the steeper the sorption isotherm in the interval in question (compare Figs. 22-28 with Tables I and II).

No dependence of the choice of relative humidity for best restoration on the freezing rate prior to freeze-drying was observed. Apparently such structural changes as are introduced to differing extents, depending on the rate at which tissues are frozen, do not affect the nature of the dependence of plastic properties on water activity.

It is interesting, where taste panel studies could be correlated with the results of the less subjective laboratory experiments, that the panel placed the best processing humidity a little higher than did the other method.

Specifically, carrots, observed in the laboratory, restored best when compressed (1) upon resorption to 50% or 60% R.H. at 25°C., (2) on desorption to 30%, 40% or 50% R.H. at -10°C. The panel preferred, by a small margin, the carrots compressed (1) after resorption to 70% R.H. and, (2) after desorption to 50% R.H. Possibly a visible restoration conceals a loss of texture detected only by sensory evaluation.

Peas, similarly investigated, restored best after compression following resorption at 70% R.H. (judged by various objective measurements). The taste panel, however, rated the material processed at 80% R.H. superior to that processed at 70% or 60% R.H. Peas desorbed to 30%, 40% or 50% R.H. at -10°C. prior to compression at 25°C. were rated, after restoration, equally acceptable to the panel, correlating well in two of the three conditions with objective measurements.

The taste panel rated carrots and peas processed by limited freeze-drying better than those processed by conventional freeze-drying and humidification. It would be most interesting to know whether or not this preference could be reproduced and whether or not the panel would rate material processed by spray remoistening lower yet. A discussion of the reason for the improved performance would be beside the point unless a further series of taste panel tests were conducted.

The restoration of the beef at 40% & 50% R.H. (Table I) and at 10% R.H. (Table II) was judged to be good. Neither raw nor cooked beef, however, restored so excellently as to warrant a full scale taste panel comparison of processing following resorption with processing after direct desorption at the same time. The impression was gained from the laboratory studies that the beef prepared by desorption was less damaged than that prepared via the fully freeze-dried state. It would appear that a total dehydration introduces undesirable changes not fully reversed by resorption. The matter deserves a further more detailed study.

Shrimp were readily prepared for compression both by resorption and by direct desorption and presented an especially interesting pattern of behavior when compressed following exposure to humidities lower than those providing best restoration. Exceptionally tender shrimp were produced, probably by fragmentation of the muscle tissues. Compression at lower humidities yielded shrimp judged soft rather than tender, partly no doubt because these materials expanded on restoration to volumes exceeding the original values.

Neither cabbage nor peaches could be processed by direct desorption since collapse ensued during limited freeze-drying at -10°C. Peaches collapsed during limited freeze-drying even at -30°. Obviously, since peaches did not collapse on freeze-drying at room temperature the ice interface temperature must have remained very low through the drying. Thus, it may be reasoned that a limited freeze-drying could be conducted at a temperature between -30° and -10°C. with a sufficiently low condenser temperature to permit a true limited freeze-drying without collapse, yielding material of the desired water content and conserving other volatiles. Further studies should be conducted on this subject.

Both cabbage and peach restored best when compressed after resorption to 30% R.H. but neither material offered a completely satisfactory recovery. Without doubt these two foods posed the greatest problem. Neither tissue possessed the resilience of the carrot despite the similar composition, the tissues consisting in each case of a cellulosic framework filled with aqueous sugars.

Probably the differences reside in the reactions of the fibrillar cell structures to impressed forces. Evidently these structures are reversibly deformed in the carrot, i.e., new stable configurations are not available. Probably, in the peach especially, one conformation is as stable as another, i.e., a cell wall, once folded, assumes a structural arrangement devoid of strain. Very likely the key to the question is in the way in which fibrils in the wall are cemented together. Further studies based on the selective digestion of hemicellulosic and pectinic substances are indicated. A detailed electron microscopic study of the cell wall structure is also strongly indicated.

5. Cytological Investigations

(a) Carrots.

It would seem that the cytological studies on carrots were of value for several reasons and served the following purposes.

First, the form and extent of the structural damage is shown to depend on the number of successive treatments. Second, the extent of the damage is seen to vary with the way that any one treatment is conducted.

Thirdly, and perhaps the most important, it is demonstrated that the very extensive cytological damage observed can be detected in carrot tissues wholely acceptable to a trained taste panel. It would be most interesting to conduct cytological studies with carrots rejected for various reasons by the panel.

Lastly, it was quite evident that direct embedding of freeze-dried material in wax was by no means successful; these

carrots were very difficult to section properly. Tissues proved to be too brittle; when sections were obtained, they were very difficult to clear and stain, tending to disintegrate. Unfortunately, techniques for the direct examination of freeze-dried specimens have not yet been worked out. Some additional efforts will be required before the method assumes the character of an established routine, by which different tissues are processed equally well.

(b) Peaches.

The cytological study revealed little structural damage in peaches rendered very resistant to rehydration by freeze-drying. Apparent damage was absent even in peaches freeze-dried, compressed and restored where restoration was so slow and yielded such poor recovery. The absence of visible damage combined with the poor recovery contrasts sharply with the case of the cooked carrots where the damage to the structures was so readily seen in materials recovering so very well.

The reasons for the failure of the peaches to recover, after compression, might best be sought in the behavior of the soluble sugars and in the properties of the pectin and the hemicellulose fractions. Neither the properties of sugars nor of the cellulosic framework sufficed to explain the difficulties encountered with peaches.

(c) Peas.

The losses in texture correlate to some extent with the observations made under the microscope. The very good recovery after compression at high relative humidities to 300 p.s.i. or to 2000 p.s.i. can be seen in the micrographs obtained from rehydrated, fixed, embedded and sectioned materials. The good restoration is also reflected in the results of the subjective laboratory experiments, and in the judgment of the taste panel.

The preservation of both intercellular relationships and intracellular systems is demonstrated in the micrographs. Cell walls and starch grains are very clearly visible after compression, restoration, fixation, etc. The detail with

which structure can be studied permits the conclusion that the higher pressure (2000 p.s.i.) results in more extensive damage than does the lesser pressure (300 p.s.i.). Unfortunately there was no opportunity to seek confirmation by a taste panel.

The moderate success with which freeze-dried material was embedded directly in wax, sectioned, and stained was also encouraging. It afforded a direct comparison of the structures resulting from slow freezing and from rapid freezing after freeze-drying and allowed a good demonstration of the different extents to which cellular structures, differently frozen, must recover (after further treatment, moreover) to yield the same final appearance.

Probably, with the aid of the microscope, the best conditions for processing the peas could be chosen with greater accuracy.

(d) Shrimp.

Such extensive fragmentation was seen in freeze-dried specimens cut after direct vacuum infiltration with melted paraffin that the results must be discounted. The method in the form in which it was used is not applicable to freeze-dried shrimp.

In contrast, the procedure by which freeze-dried shrimp were rehydrated, fixed and embedded appeared to provide a clear and reliable indication of the effects of compression. The slight tendency for cracks to form across the fibers was noted only in compressed and restored material.

Unfortunately the microscope did not distinguish between the freeze-dried, compressed and restored material frozen at different rates prior to freeze-drying, as did the taste panel. Perhaps, in shrimp, there is a basis for tenderness best explained at this stage in terms of submicroscopic composition and configuration.

6. Solvent Extraction Studies.

(a) Raw beef.

Since the removal of the lipids by solvent extraction did not seem to alter the way in which the raw freeze-dried beef reacted to remoistening, compression and rehydration it would appear: (1) At the lower humidities fibers retain an elasticity which is "locked in" first by redistribution of fats, secondly by mechanical entanglement, lastly by final drying; (2) where compression is conducted at high relative humidities the protein matrix is so soft it deforms without resistance and achieves a new stable conformation; (3) the native proteins are readily rewetted despite the presence of fats; (4) where a sample is unable to accommodate and relieve an imposed strain during compression, the fibrous structure undergoes spontaneous rearrangement on rehydration; which is to say simply, that the problems encountered in the case of raw beef are not to be traced to the behavior of the fats.

(b) Cooked beef.

To explain the behavior of the cooked beef in a manner consistent with the reasoning just applied to raw beef, it must be concluded that the denatured proteins in the cooked beef exhibit a much greater physical resilience, even at higher water contents, and that compression, even at higher humidities, fails to bring neighboring elements into such intimate and extended contact that a permanent adhesion is achieved in the absence of the fats.

(c) Cabbage.

The observation that the effect of compression on the water-extracted cabbage varies with the freezing rate prior to freeze-drying suggests (1) that the degree to which the cellulose structures are damaged depends on the freezing rate, (2) that solvent extraction may provide a means of distinguishing the different effects of different processing treatments on other tissues where the study of the whole tissue did not.

(d) Carrots.

The remarkable recovery of the extracted carrots, after compression at the lower humidities (in which conditions

unextracted material fragmented), suggests the extreme importance of the state of the sugars. One is led to conclude that, at too low a water content, the sugar residues acquire brittle or glass-like properties and, when compression causes such material to shatter, embedded elements fracture simultaneously. In the absence of the sugars, the filamentous elements comprising the cell wall evidently undergo reversible displacements with respect to one another upon compression.

It would appear also, since the extracted material lost the ability to restore after compression at high relative humidity, that the sugars are necessary at higher relative humidity to prevent the compression from giving the moist structural elements a "set"; possibly the sugars prevent one cellulosic element from adhering to another.

(e) Peaches.

Presumably the compression of the cell wall structures by themselves proceeds with least permanent damage, by coincidence, at 30% R.H. as does the compression of the whole peach containing also the sugar-rich soluble materials.

Evidently the compression at higher humidities allows a dispersion of the sugar residues such that cellulosic materials are caused to adhere (except at 80% R.H., where, one must conclude, the fibrous elements are too wet to accept a "set" as a result of compression).

(f) Shrimp.

The reasoning applied to cooked beef can be used to explain the observation that the solvent-extracted shrimp demonstrates best restoration after compression at 70% and 80% R.H. while the best humidities for whole shrimp are 50% and 60% R.H. It is likely that denatured proteins resulting from cooking retain, after freeze-drying, a sufficient elasticity that neither permanent deformation nor permanent adhesion result from compression even at 70% or 80% R.H. The presence of the lipids, on the other hand, would seem to permit an irreversible adhesion at 70% R.H., and higher, and to require compression at the lower water activity where the fiber proteins are still less amenable

to distortion and the lipids are, consequently, unable to effect widespread fiber-fiber contact.

7. Scanning Electron Microscopy.

(a) Carrots.

The scanning micrographs show very clearly (1) that there are some definite narrow spaces left in the carrot after compression, thus helping to explain the ready separation of the components on recovery, (2) that there is considerable room for bending during compression such that fracture is avoided. It is doubtful if these could have been seen other than by direct observation.

Successive cell walls are distinguished though these are tightly compressed together. Probably the fracture process employed to generate the cross section progressed in a stepwise manner. The method would seem to have very considerable potential in the examination of freeze-dried foods. An understanding of the technique is, however, required to permit the recognition of artifacts arising during specimen preparation.

(b) Peaches.

The use of the freeze — freeze-fracture — freeze-dry sequence serves well to prepare peaches for examination by scanning microscopy. The scanning micrographs permit the distinction between intracellular and extracellular spaces in a most unambiguous manner. The viewing of "stereo pairs" is particularly useful, justifying the effort required to obtain the necessary photographs.

The extreme difficulty encountered in the rehydration even of uncompressed material, however, implies an obstruction which is not observed. Most logically this consists of a boundary created upon rehydration, e.g., a very concentrated sugar syrup or a swelled pectin gel. Probably the examination of material frozen, freeze-dried, and rehydrated could be conducted in the scanning electron microscope after a second freeze-drying (or a freeze substitution) carried out in such a way as not to permit a

renewed redistribution of the solutes in question. Certainly further observations on freeze-dried peaches, especially after compression, should be made with the scanning electron microscope.

(c) Peas.

The scanning microscope appears to be particularly effective in revealing three dimensional arrangements in both uncompressed and compressed freeze-dried peas. The presence of a "freezing pattern" is more clearly seen than it is by thin sectioning, for instance.

In the compressed peas the low temperature fracture method of preparing a surface appears to be especially useful. The small spaces visible between the solid components most likely represent artifacts of the fracture process and, bear evidence to the consistency of the moist material. Consequently it would seem that the best restoration is achieved where the components are less than fluid at the microscopic level.

Frequently the structures within the compressed material are seen to undergo multiple folding. Here and there, however, it appears that starch grains burst through cell walls. It is also demonstrated that a continuous system of empty spaces is frequently retained after compression. Semiquantitative estimates of "porosities" could presumably be obtained after sufficiently detailed examinations of compressed freeze-dried materials in the scanning electron microscope. Comparisons of rates and degrees of recovery and of other properties of compressed freeze-dried products could be made with reference to such measured "porosities."

CONCLUDING REMARKS

In pursuit of the objectives of this study we chose to employ (a) different freezing rates, (b) different freeze-drying procedures, (c) different relative humidities prior to compression, (d) different compression procedures. Since each of these factors was examined on the premise that restoration could best be studied with reference to water activity prior to compression it will be useful to draw some conclusions concerning that premise.

In the light of the finding that relative humidity constitutes a good basis for determining processing parameters, some practical aspects of the various steps involved in the production of compressed materials are examined.

1. Description of the Potential Behavior of Compressed Materials in Terms of Water Activity Prior to Compression.

It is clear, both from a theoretical consideration and from the results presented in this report that preparation for compression can indeed be formulated in terms of relative humidity, that is, in terms of water activity (a_w) established, in defined circumstances, in the food before it is compressed. Experiments sufficient to support this conclusion could be conducted in any laboratory without practical difficulties.

A determination of the water content in terms of the activity of the water — the derivation of a sorption isotherm — then allows the definition of a best moisture content for compression. The water activity yielding the water content providing best compression will depend only on the sample temperature during the sorption measurements and on whether the latter required the desorption or the resorption of the sample. The direct determination of best moisture contents by the addition of various quantities of water to dry materials in conditions where the humidity was not controlled would, in contrast, be objectionable.

On the basis of these findings it is evident that different foods may be "matched" with respect to their dependence on water activity for best compression. Where a composite material is to be freeze-dried, humidified, and compressed, the mere addition of the right amount of water to the fully freeze-dried mixture, based on the right amounts of water needed by the components, may well fail to produce a moist foodstuff suitable for compression. The choice of components, on the other hand, each having its best performance after compression at the same humidity insures the best

compression of the mixture at that humidity, with best restoration after final drying, storage and rehydration. The use of the water activity concept allows the formulation, on a rational basis, of mixtures destined for compression.*

(A two-step procedure, less direct than that just described, would be required if foods prepared by limited freeze-drying were to be "matched" for compression. Foods freeze-dried in a mixture by sublimation and direct desorption to a defined relative humidity at, for example, -10°C. would each maintain their own best moisture contents for compression only as long as the temperature remained unchanged. A change in the temperature of the isolated mixture could cause water to redistribute, depending on the extents to which the activity of the water present in each component tended to increase on warming. But such changes could be measured quite easily in separate experiments.)

2. Ways to Bring Foods to the Desired Water Activity before Compression.

Several different experimental procedures for the production of freeze-dried foods exhibiting defined water activities appear to be ready for use in pilot-scale situations.

Foods can be prepared for compression by extensive drying followed by rehydration (humidification) in an atmosphere or vacuum of controlled relative humidity generated with liquid water or with ice. These operations could be carried out in one apparatus, properly arranged, or in a series of interconnecting or separate apparatus.

*One cannot discuss a subject such as this without an acknowledgement to Salwin (1962) for his classic studies on the tendency for water present in freeze-dried foods to redistribute when the foods are mixed together.

Alternatively, foodstuffs could be prepared for compression by vacuum sublimation of ice followed by direct desorption (limited freeze-drying) in an atmosphere or vacuum of controlled relative humidity.

(Sulphuric acid solutions do not seem to lend themselves to use in pilot-plant or large scale operations — though they proved most useful in the laboratory. Salt solutions might, however, be of use in production of atmospheres of controlled humidity as might, for example, ethylene glycol, glycerol or sugar solutions.)

(A solution of any volatile substance could also provide the basis for a method of subliming ice and achieving a predetermined degree of desorption; its use could allow the development of an alternative process to limited freeze-drying.)

There would appear to be several advantages in the use of the more conventional freeze-drying procedures. These include the utilization or modification of existing equipment.

With reference to cost, limited freeze-drying would require the more extensive modification of existing equipment, or the construction of a new plant. Moreover, processing times are several fold longer (see Tables III and IV, pp. 30-31). One could, however, cite the slight preference for the material prepared by direct desorption noted by the taste panel. It is also likely (1) that fats will not redistribute within the product as extensively during limited freeze-drying as they might during freeze-drying at room temperature, (2) that emulsions will not be destabilized and broken during limited freeze-drying. It is also highly probable that volatile fractions will be more extensively conserved during limited freeze-drying. The advantages of the method have yet to be fully determined.

3. Humidification Rates.

Resorption velocities are high enough to be of practical interest and to justify pilot-plant studies on

the moistening of freeze-dried material, prior to compression, via vapor transfer. Since the foods themselves seemed to determine the rates of resorption, the design of the necessary equipment should not be difficult.

The rate of water uptake during humidification might be measured by continuous weighing of an entire load or of a representative part. Where the current commercial practice requires that the freeze-dried food, and the water to be added to it, each be weighed, the controlled humidification would require only the observation that the sample weight had ceased to change.

The moistening by regulated vapor transfer offers other advantages. The outermost portions of the freeze-dried food will not be overmoistened (and hence, possibly, damaged) prior to a final equilibration. Similarly, soluble components will not be leached from the portions first remoistened and carried into the interior or across the product.

4. Rate of Final Drying.

The desorption of water from the moist compressed product was found to proceed quite rapidly, though not perhaps as rapidly (nor as completely) as did the drying of similarly moist but uncompressed control samples.

(A consideration of the times taken for a particular sample to freeze-dry to a very low moisture content, to humidity to a desired moisture content, and to dry to a low moisture content again, after compression, suggests the usefulness, in larger scale operations, of a multiple unit complex. It would consist of several freeze-drying chambers, each with its own condenser, each drying chamber/condenser unit being linked to a common water vapor generator, capable of producing water vapor of a defined water activity at a certain temperature. The sequential loading of the several freeze-drying chambers combined with the sequential use of the common water vapor generator and the simultaneous defrosting of the respective condensers, might prove very economical.)

5. Rates of Limited Freeze-Drying.

The rates of limited freeze-drying were found to be high enough in some cases to warrant serious consideration of the method. A study of the economics of the process with respect to the possible production of a superior product appears to be justified.

The rates of the final drying process were found to be high enough to warrant the use of the same chamber used for the limited freeze-drying.

6. Scaling-Up of Limited Freeze-Drying.

Heat transfer proceeds principally by conduction across and convection within the water vapor present between samples and supporting and enclosing surfaces. With the small temperature differences (5 to 20°C.) established in limited freeze-drying thin loadings are highly desirable. Thus, single layers of shrimp, double layers of carrots and 3 or 4 layers of peas would result in temperature gradients falling, during drying, to 5 to 10 deg. C. per cm. These gradients are, however, established in the presence of relatively high heat transfer coefficients, the water vapor being comparatively dense in the conditions of limited freeze-drying.

Mass transfer proceeds without difficulty. Sufficiently large ducting from sample chamber to condenser results in evaporative cooling in the sample to the point where ice interface temperatures approach condenser temperatures; vapor pressure differences become very small, permitting a maximum difference in temperature between sample chamber walls and sample ice interface, approaching the difference between sample chamber and condenser temperatures.

The pumping of non-condensibles does not seem to interfere with limited freeze-drying. It appears possible to

remove from the system (1) such quantities of permanent gases as are evolved from the frozen food during freeze-drying, (2) gases introduced into the system through leaks across the various joints and gaskets. Since it should not prove difficult to construct larger systems with low or lower leak-rates per unit volume than those constructed in the course of this work, no problem should arise in the disposal of permanent gases and the simulataneous regulation of water vapor pressures.

Feedback-regulated control of sample chamber and condenser temperatures should be possible as much with a 100-pound or a 1000-pound capacity system as it is with a 1-pound capacity apparatus. Direct feedback-regulated control of temperature difference might, however, prove more economical. Thus, the temperature of the sample chamber could "cycle" with an amplitude of 1 deg. C., or more, as long as the condenser temperature tracked in phase at a constant difference (the difference is best maintained constant to a fraction of a degree) or vice versa. One should also consider the case where the condenser temperature is allowed to cycle (as it sometimes does when the condenser is mechanically refrigerated) and the sample chamber is permitted to track by mixing feedback-regulated heating with overcooling.

End point determinations can conveniently be made by the method of vapor-pressure-rise-to-equilibrium (not to be confused with the Leybold method of vapor-pressure-rise since this latter test is a dynamic method). V.p.r.t.e. is a nondestructive method when applied to limited freeze-drying (but destructive when applied in the course of warm-shelf freeze-drying). V.p.r.t.e. is, furthermore, applicable regardless of sample loading and arrangement since it involves the measurement of an equilibrium pressure. The Leybold method of vapor-pressure-rise could also be used but, since it depends on the observed rate of increase of vapor pressure after the isolation of the sample chamber, and since this depends on the sample, sample loading, and nearness to the end point, each production routine would require a separate calibration.

7. Compression in Vacuum.

Compression in the absence of air could be used to advantage (1) to speed compression, (2) to prevent the "bounce" sometimes seen after the compressive force is removed, (3) to avoid exposing the freeze-dried specimen to oxygen.

Compression in the absence of air is especially well suited to humidification, conducted in the absence of air, and vice versa. The humidification and the compression can be carried out in the absence of air in the same apparatus in which the material was freeze-dried.

The process of compression in the absence of air is also very well suited to the process of limited freeze-drying, and vice versa. Each of these processes can be carried out in one apparatus, as was demonstrated.

The benefits arising from the use of equipment in which sample transfer is avoided should be investigated on a practical basis, at the pilot-plant level.

8. The Effect of the Initial Freezing Rate.

The rate at which the foods were frozen prior to freeze-drying did not appear to determine the patterns of restoration subsequent to compression after exposure to atmospheres of various relative humidities. Neither did the freezing rate appear to determine the extent of the restoration after processing in conditions chosen to yield best restoration.

Some effects attributable to the freezing rate were, however, noted in shrimp (pp. 27 and 36). Where the freezing rate is found to determine restoration, other processing conditions aside, further studies encompassing as broad a range of freezing rates as possible should be considered.

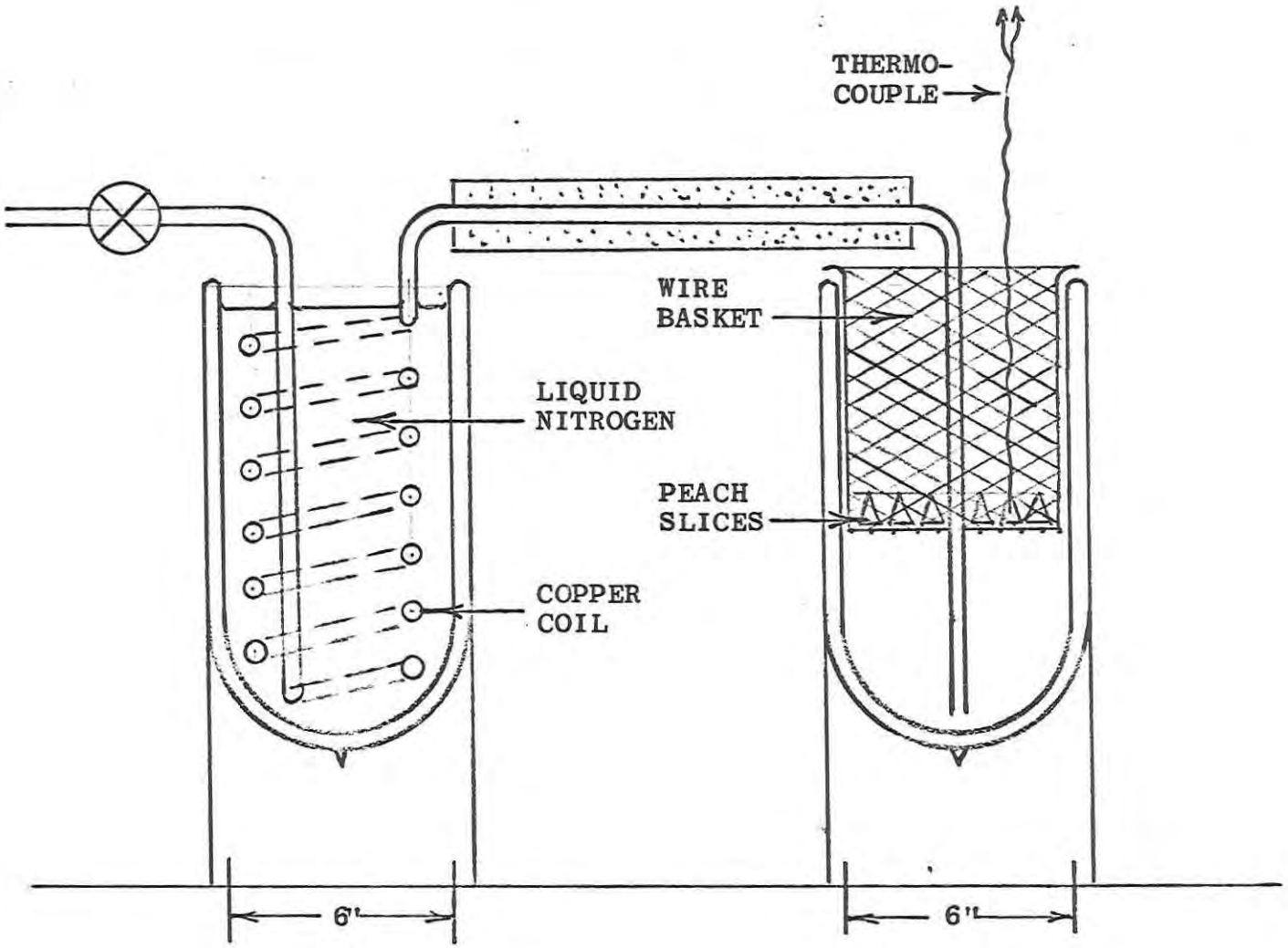


FIGURE 1. APPARATUS FOR FREEZING IN NITROGEN GAS AT -196°C.

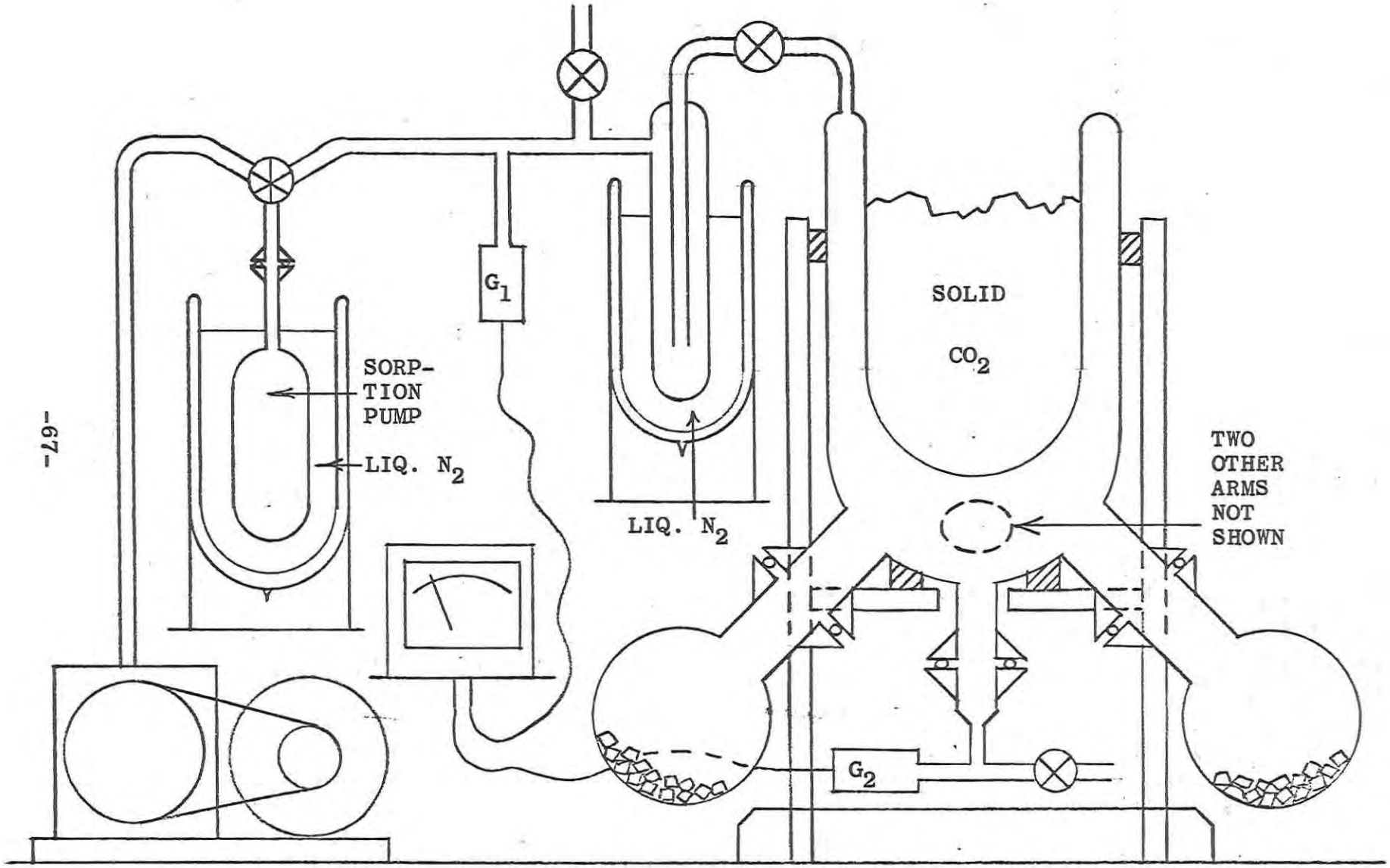


FIGURE 2. APPARATUS FOR CONVENTIONAL FREEZE-DRYING

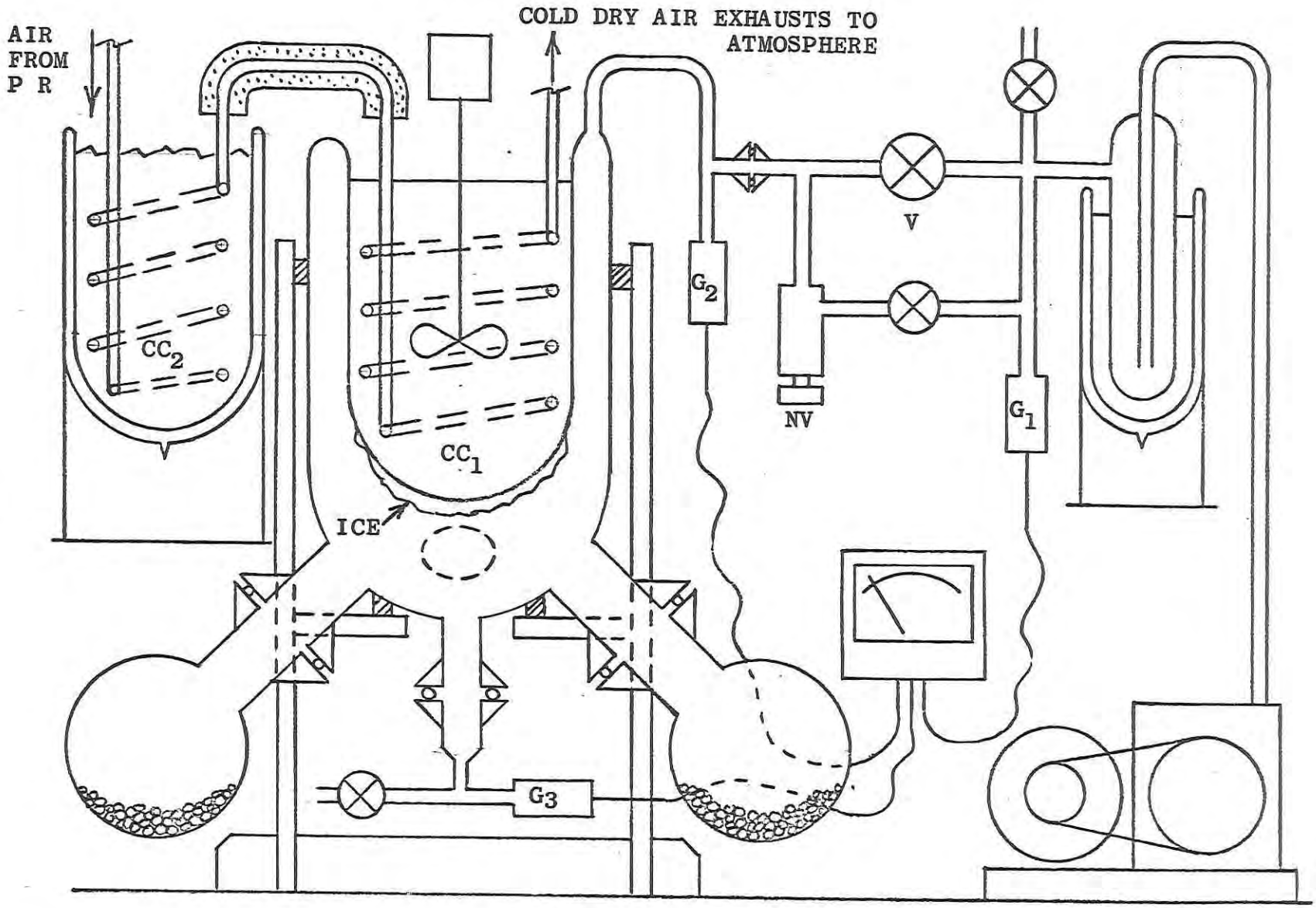


FIGURE 3. APPARATUS FOR "LIMITED FREEZE-DRYING"

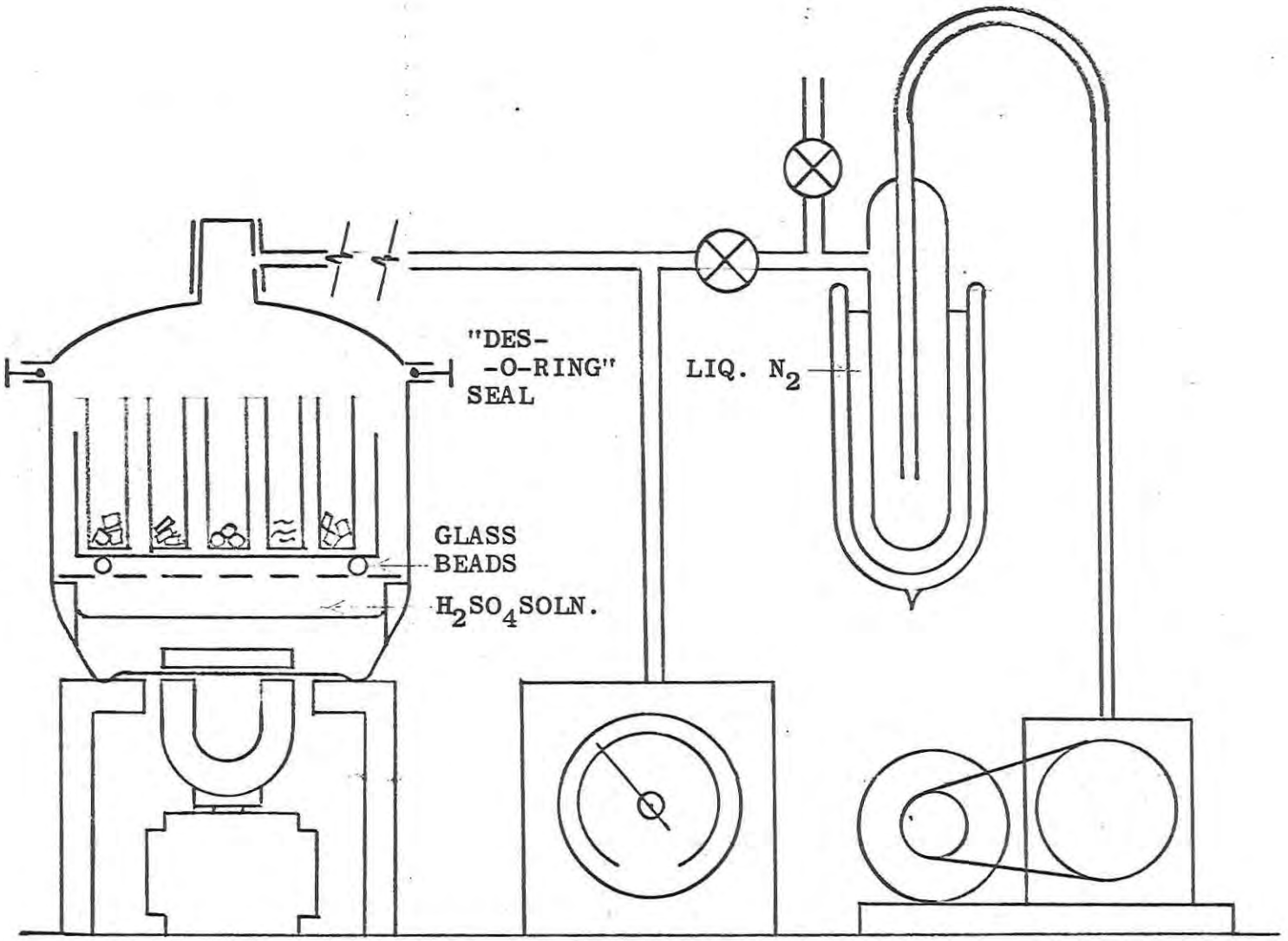


FIGURE 4. DESICCATOR PUMP-DOWN ASSEMBLY

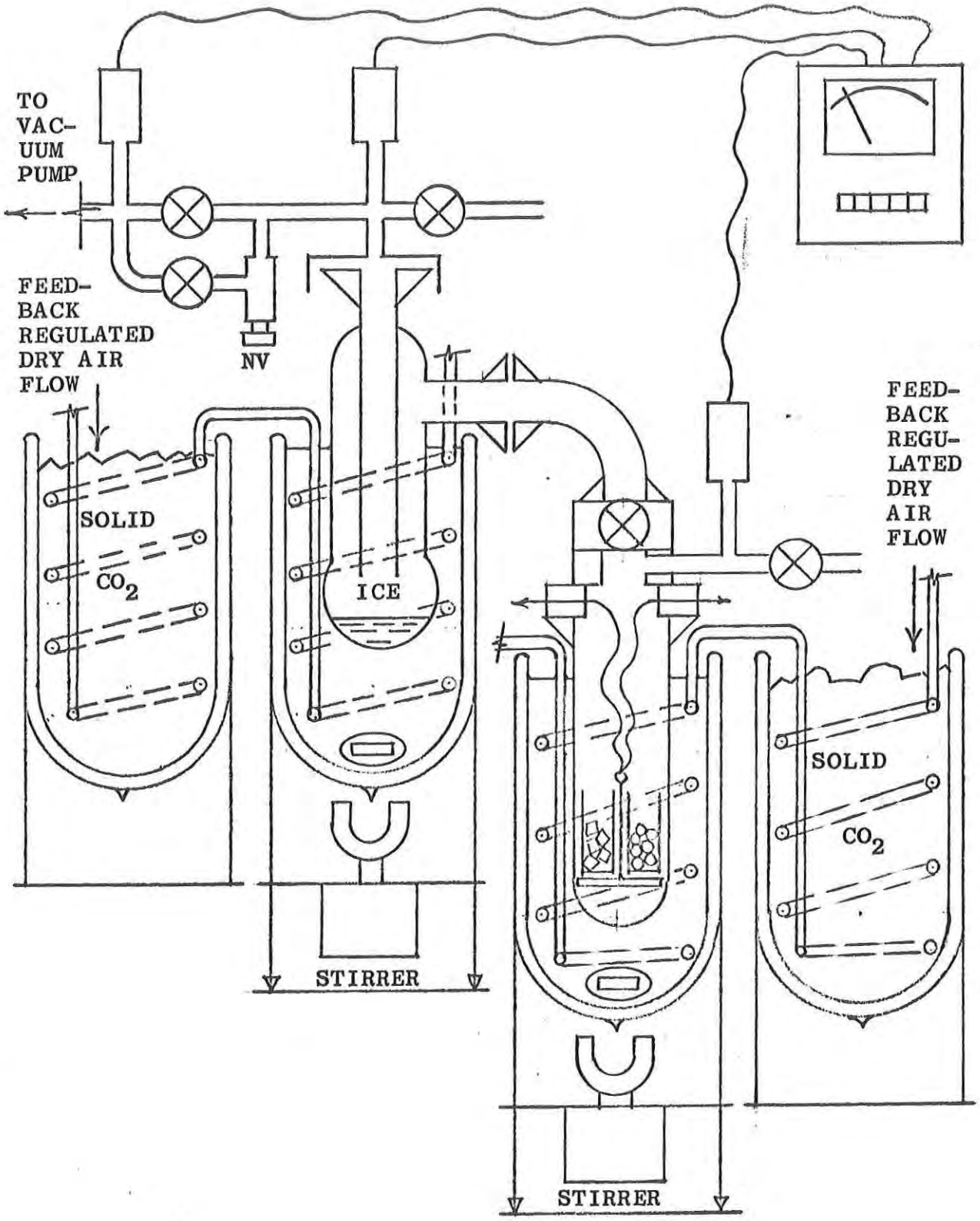


FIGURE 5. APPARATUS FOR "LIMITED FREEZE-DRYING"

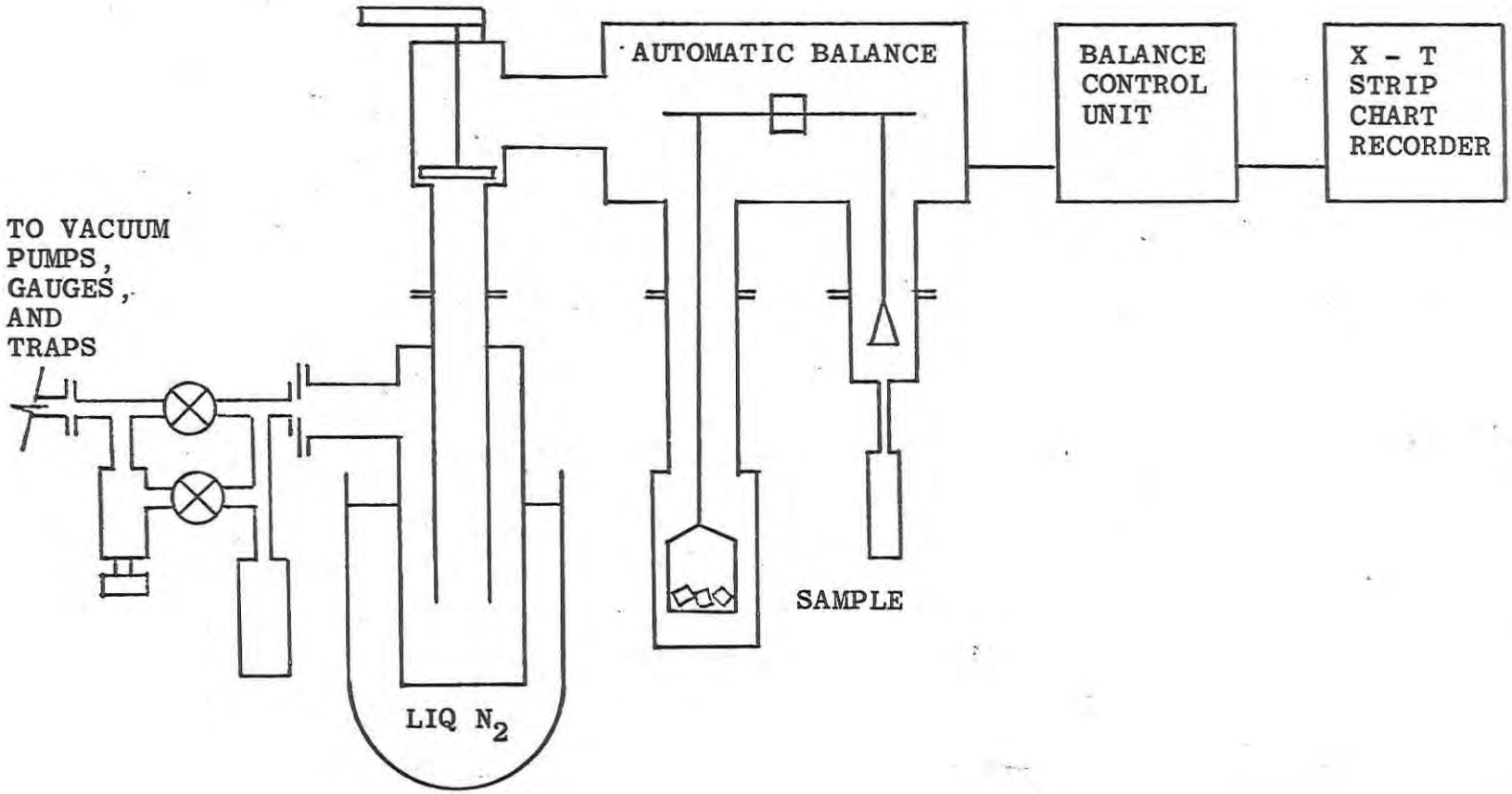


FIGURE 6. EQUIPMENT FOR THE DETERMINATION OF FREEZE-DRYING RATES

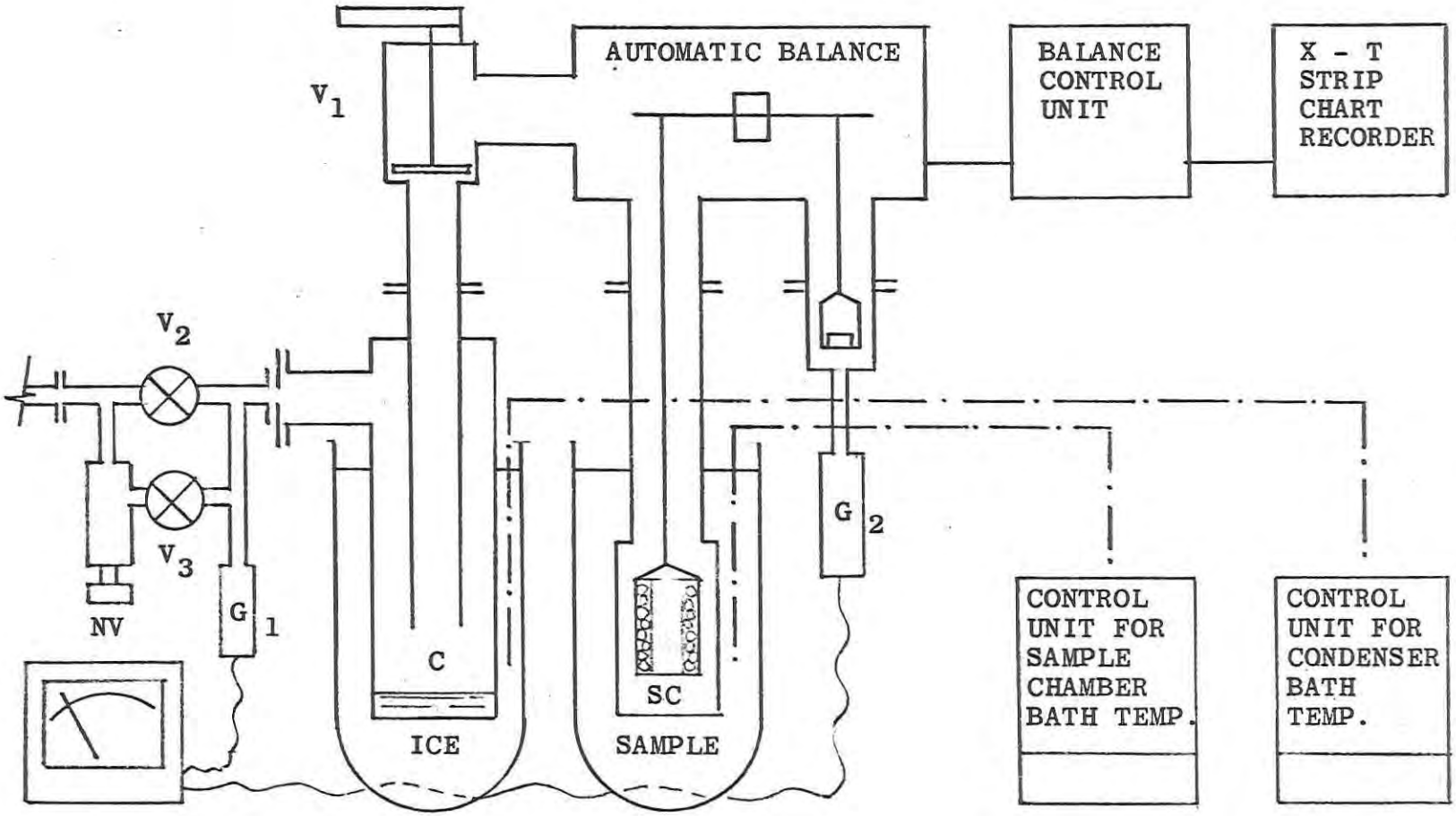


FIGURE 7. APPARATUS FOR THE DETERMINATION OF THE RATE OF "LIMITED FREEZE-DRYING"

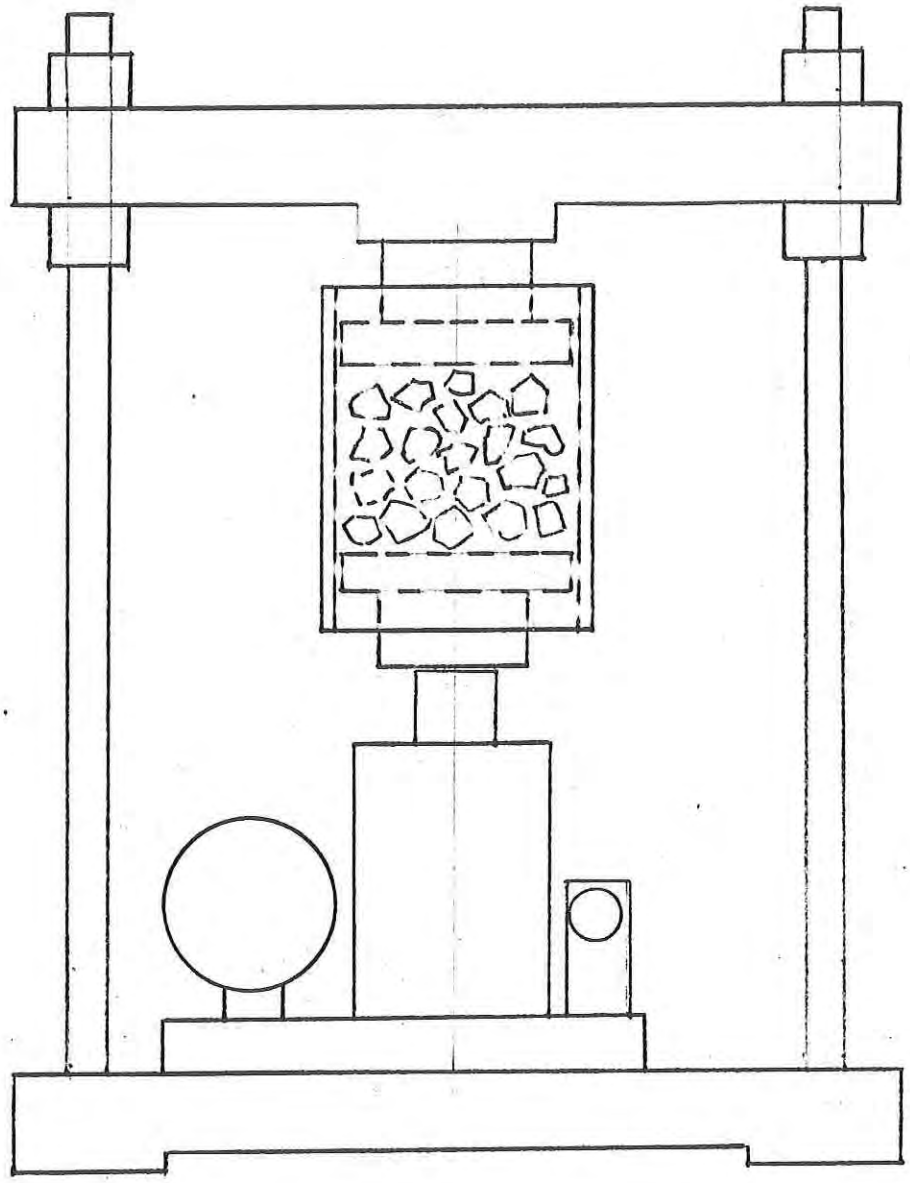


FIGURE 8. COMPRESSION ASSEMBLY

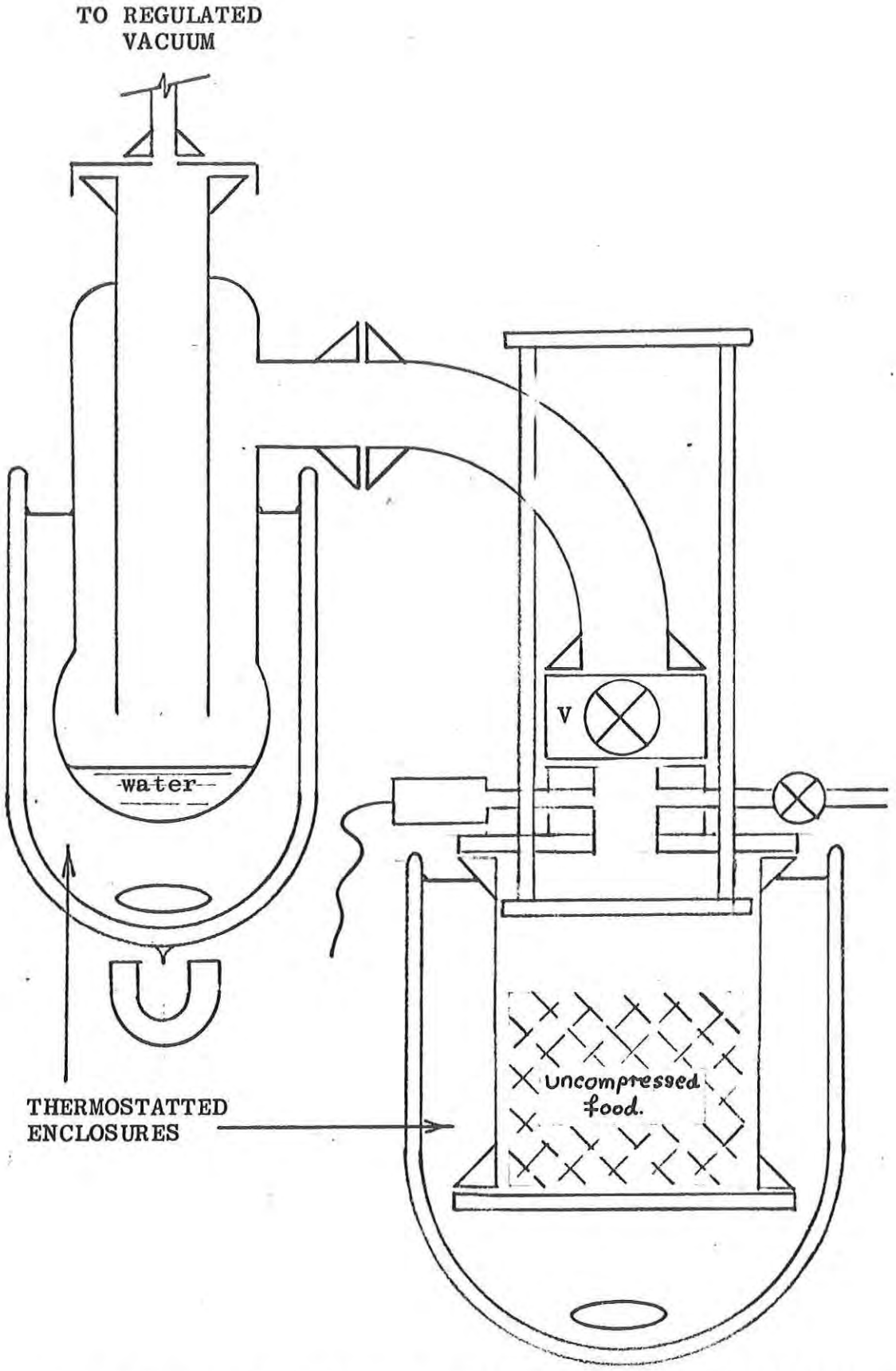


FIGURE 9. APPARATUS FOR COMPRESSION IN VACUO (AFTER HUMIDIFICATION)

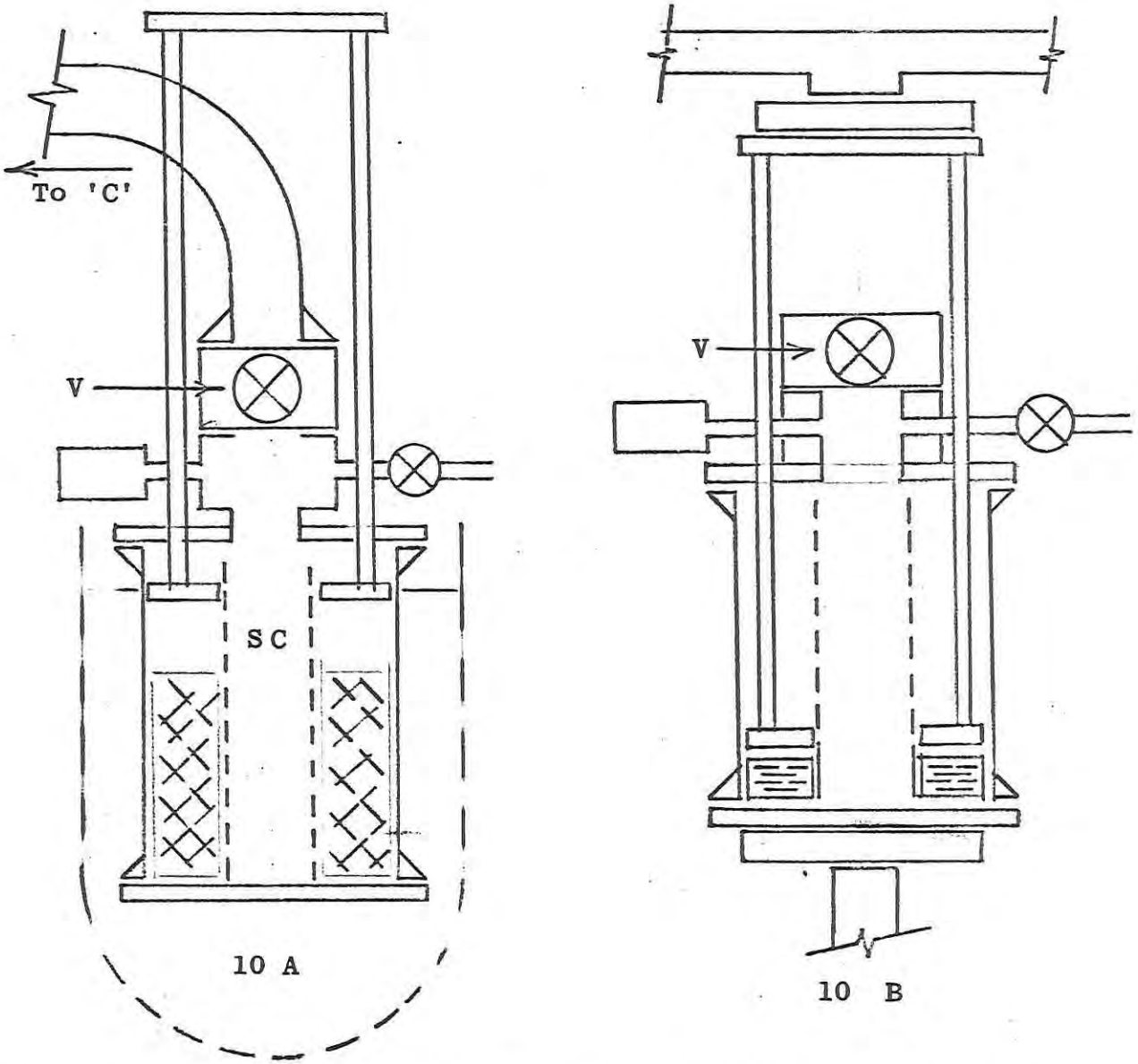


FIGURE 10. APPARATUS FOR COMPRESSION "IN THE FREEZE-DRYING APPARATUS"

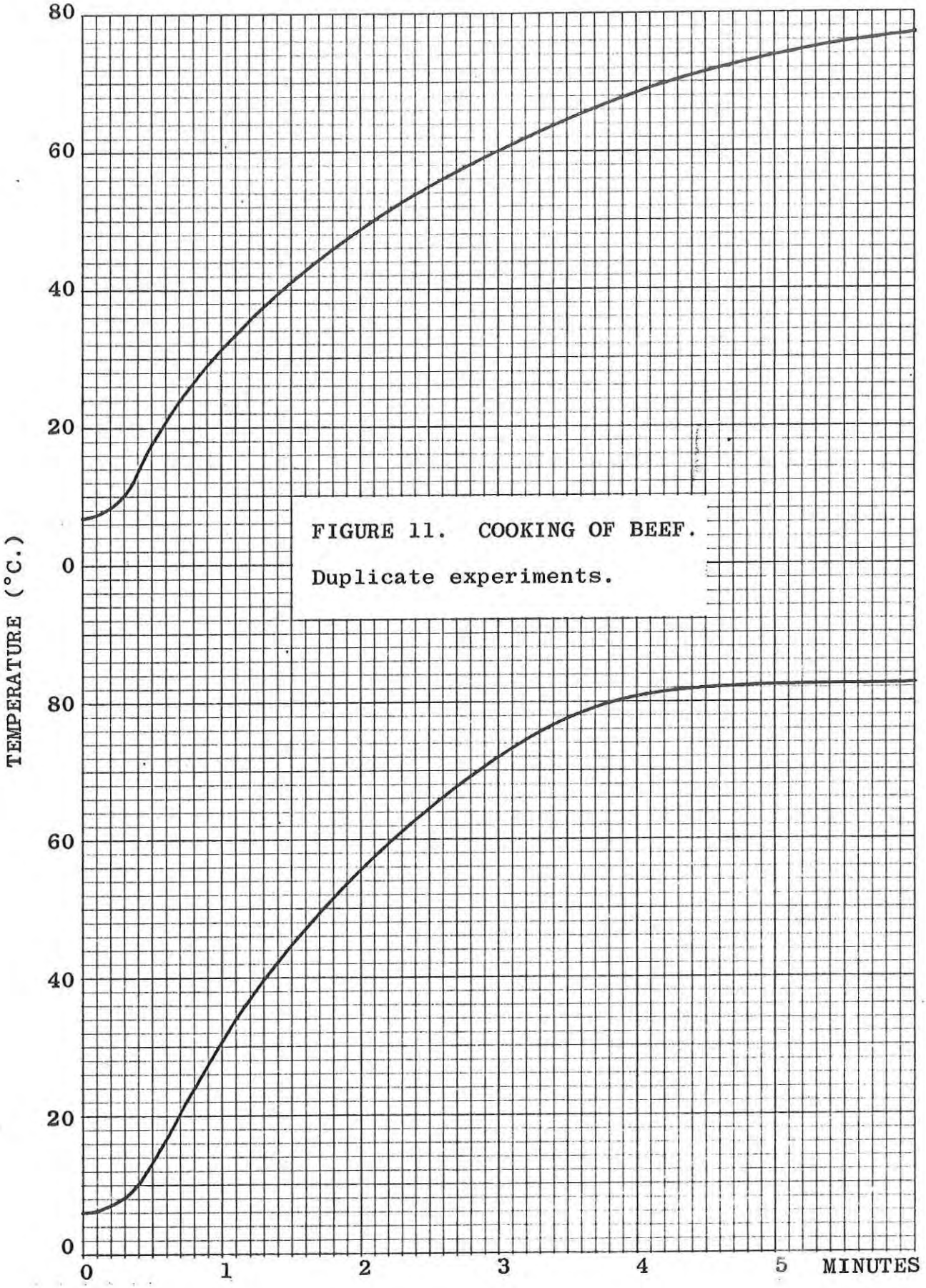


FIGURE 11. COOKING OF BEEF.

Duplicate experiments.

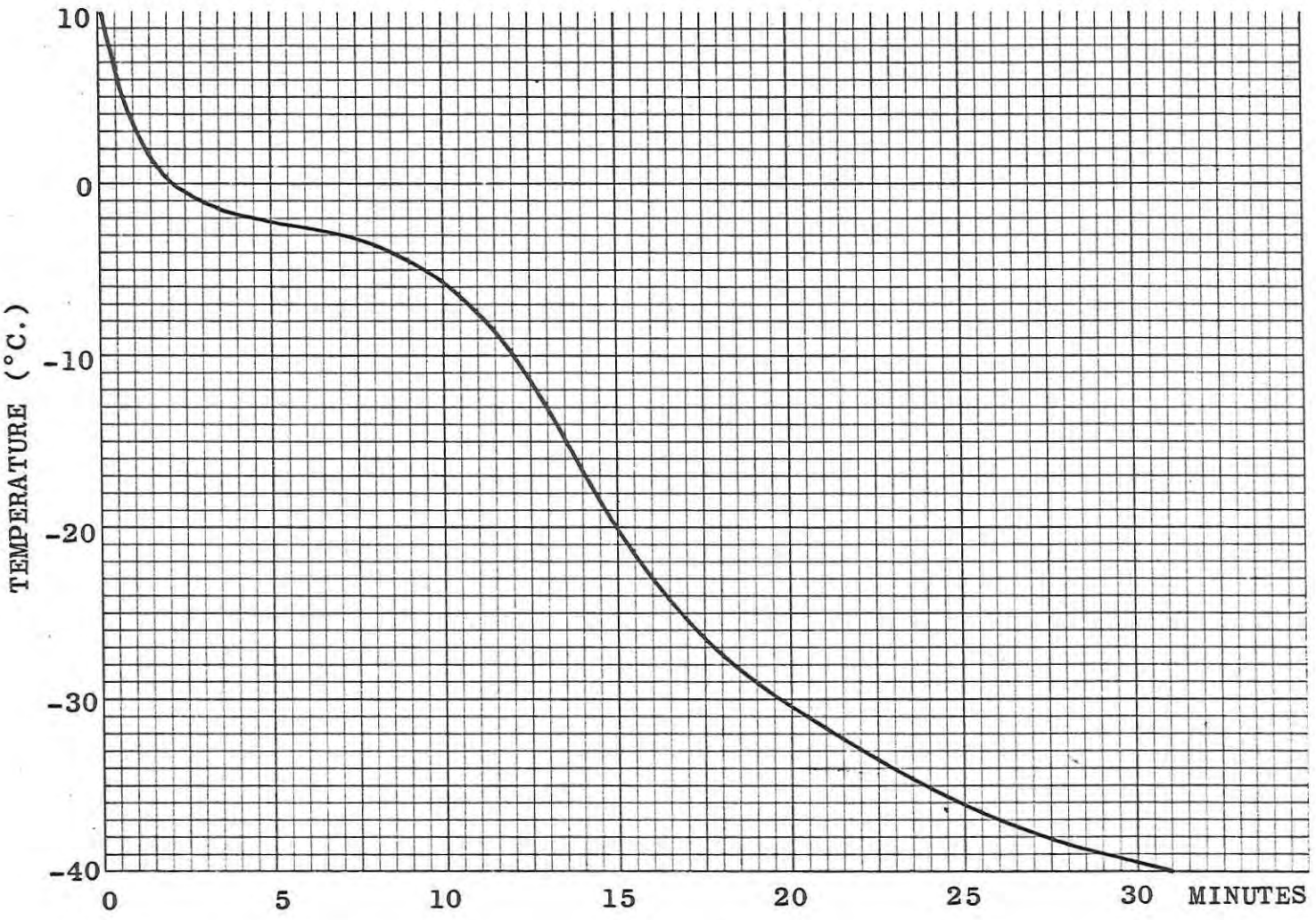


FIGURE 12. FREEZING OF RAW BEEF IN STILL AIR AT -40°C.

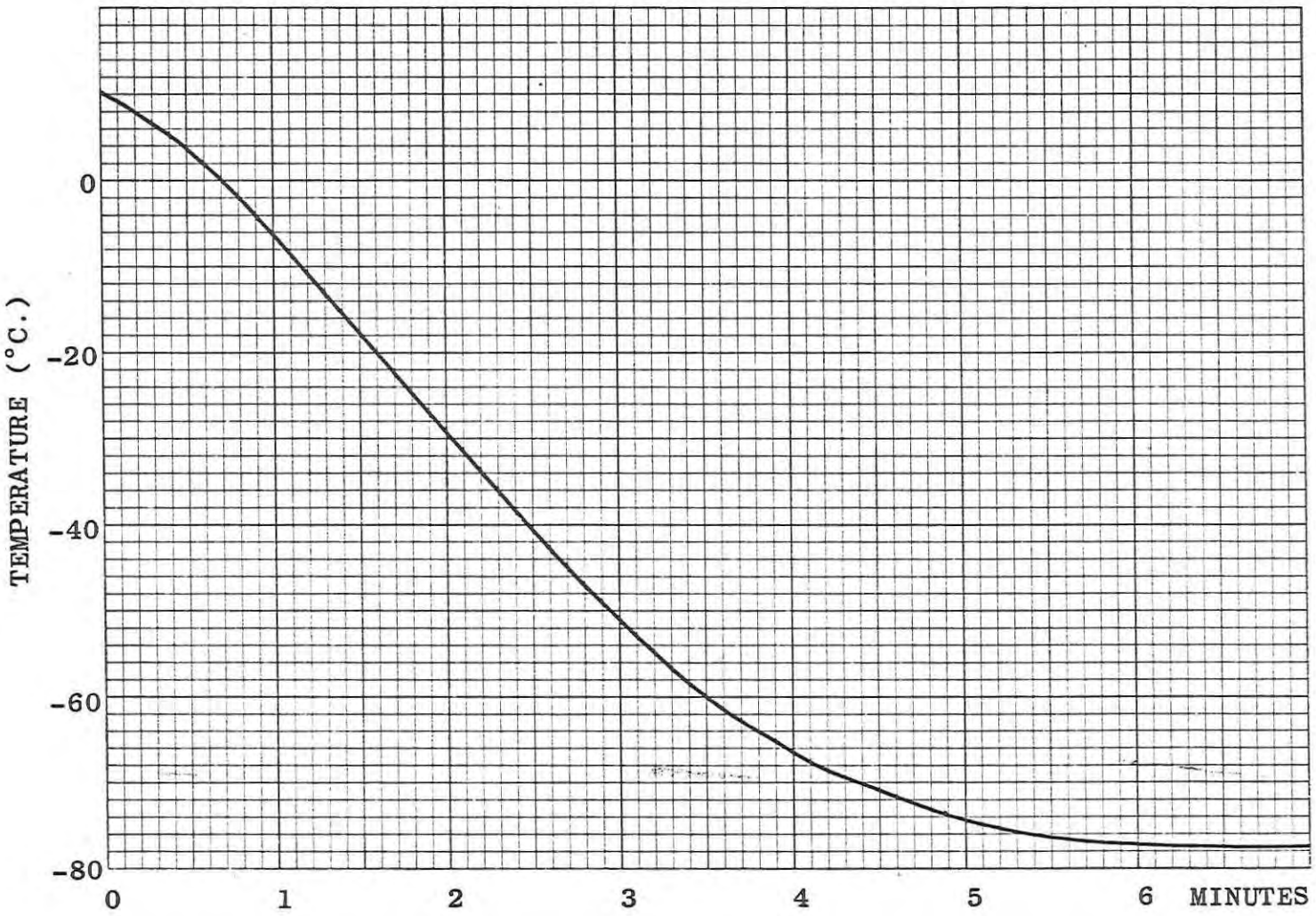


FIGURE 13. FREEZING OF RAW BEEF IN SOLID CARBON DIOXIDE

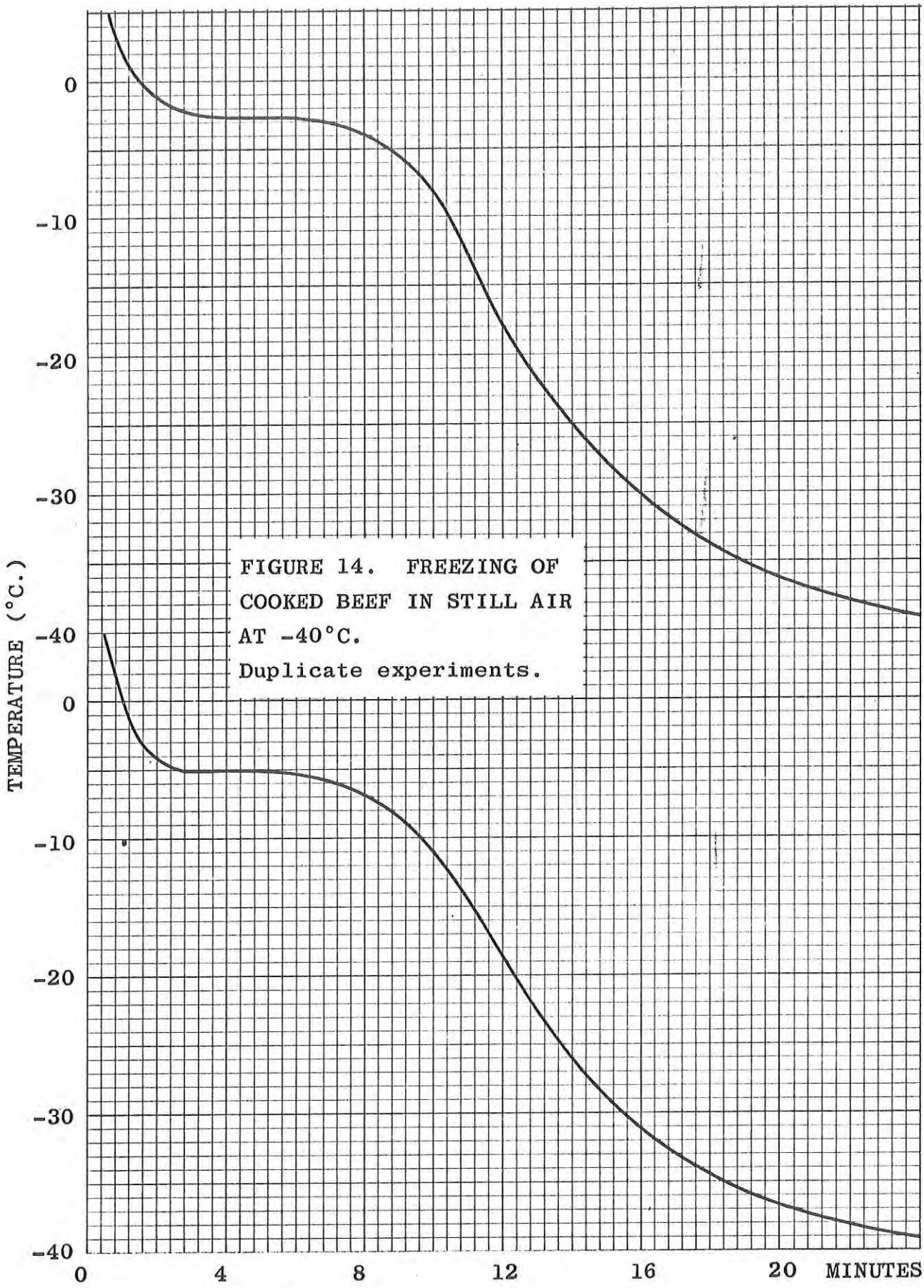


FIGURE 14. FREEZING OF COOKED BEEF IN STILL AIR AT -40°C.
Duplicate experiments.

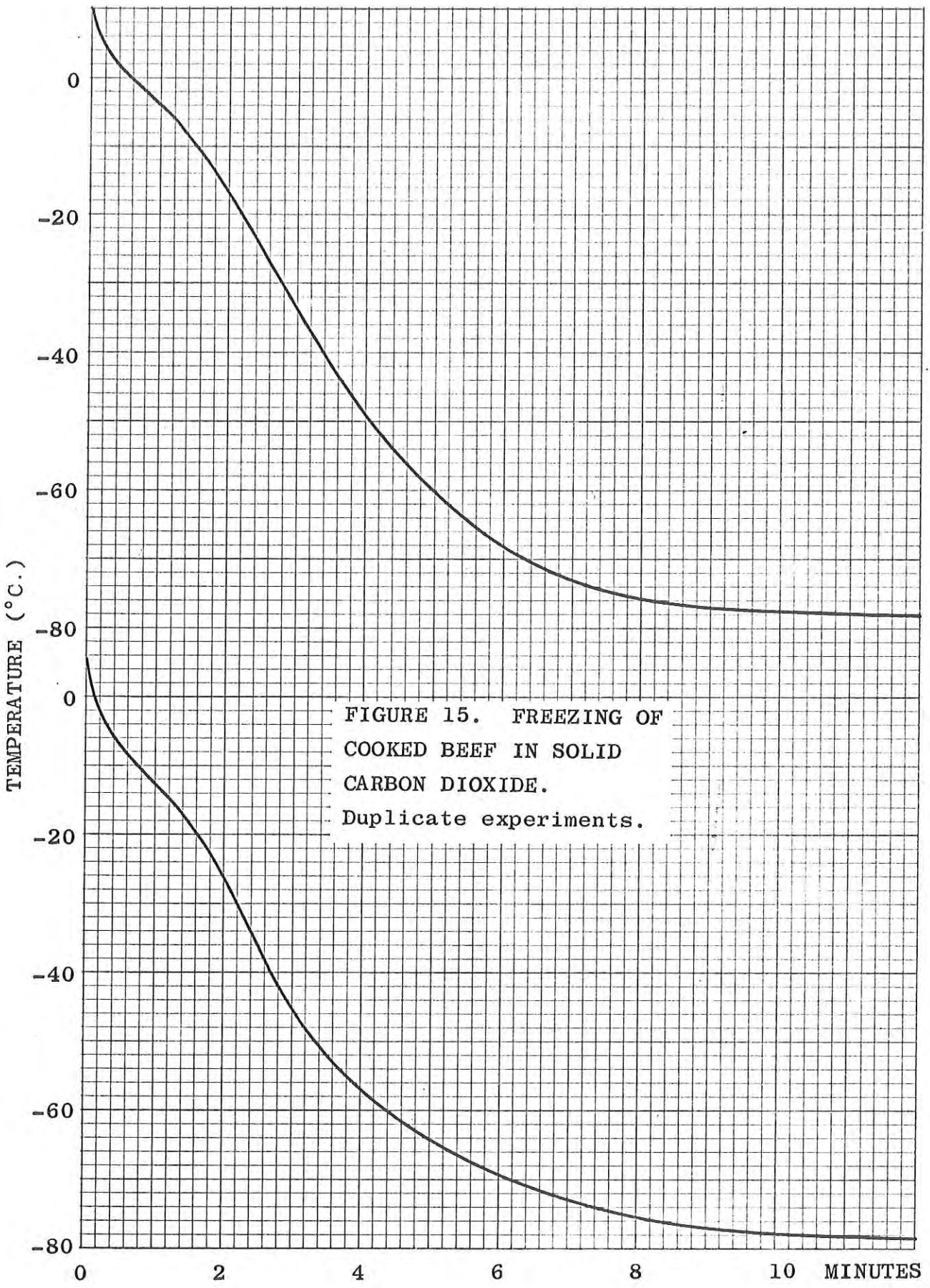


FIGURE 15. FREEZING OF COOKED BEEF IN SOLID CARBON DIOXIDE. Duplicate experiments.

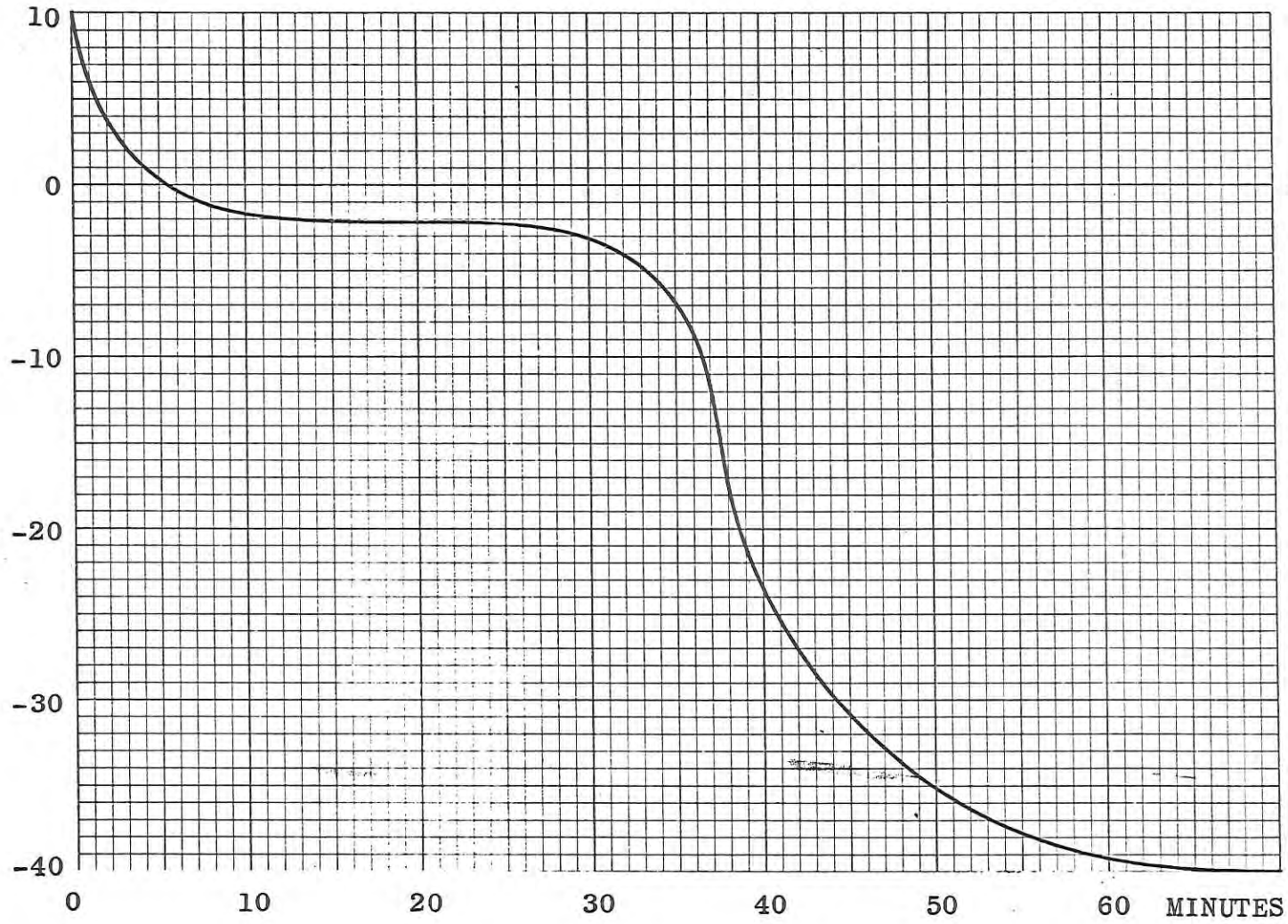


FIGURE 16. FREEZING OF COOKED CARROTS IN STILL AIR AT -40°C.

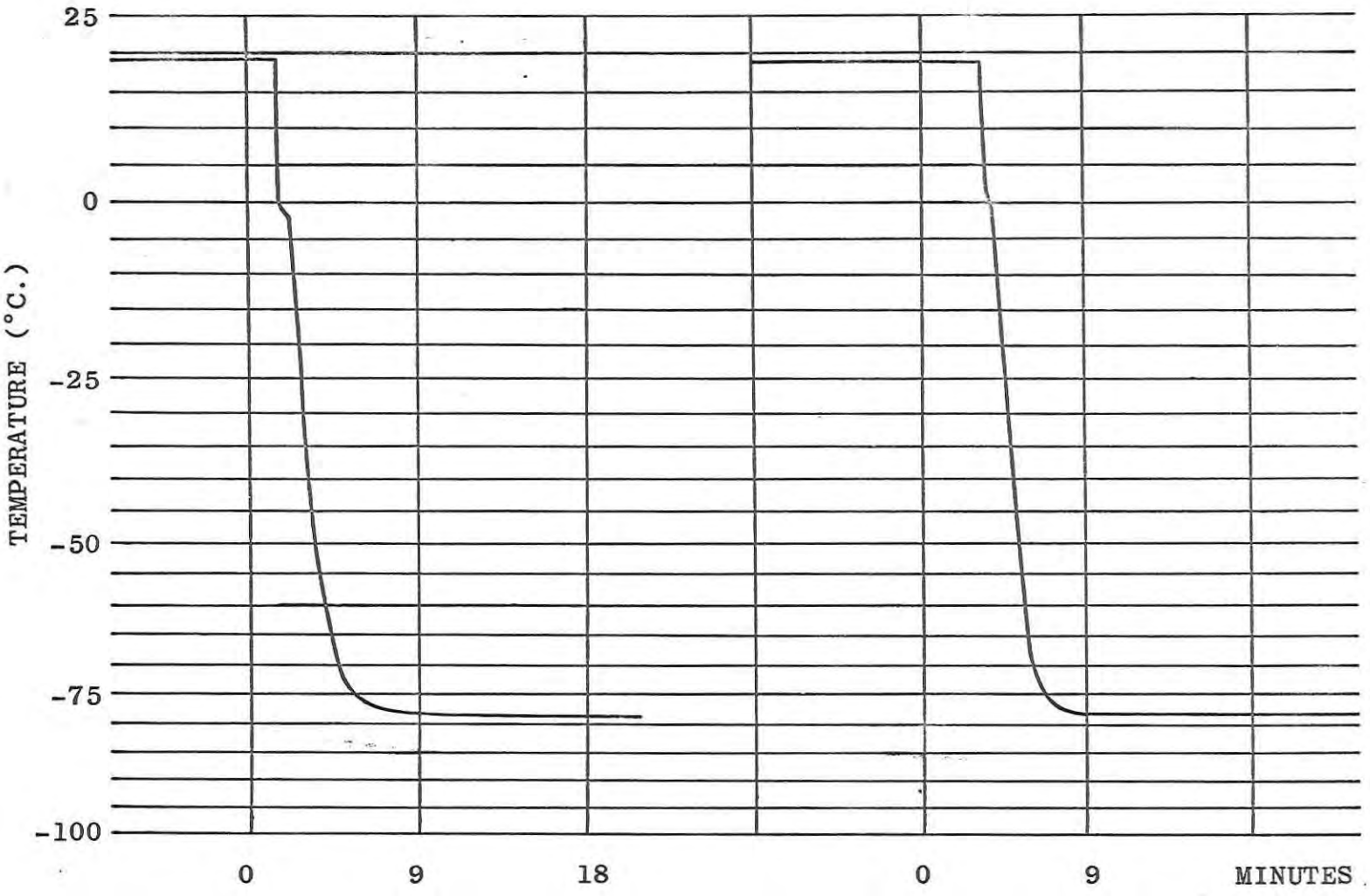


FIGURE 17. FREEZING OF COOKED CARROT CUBES IN SOLID CARBON DIOXIDE

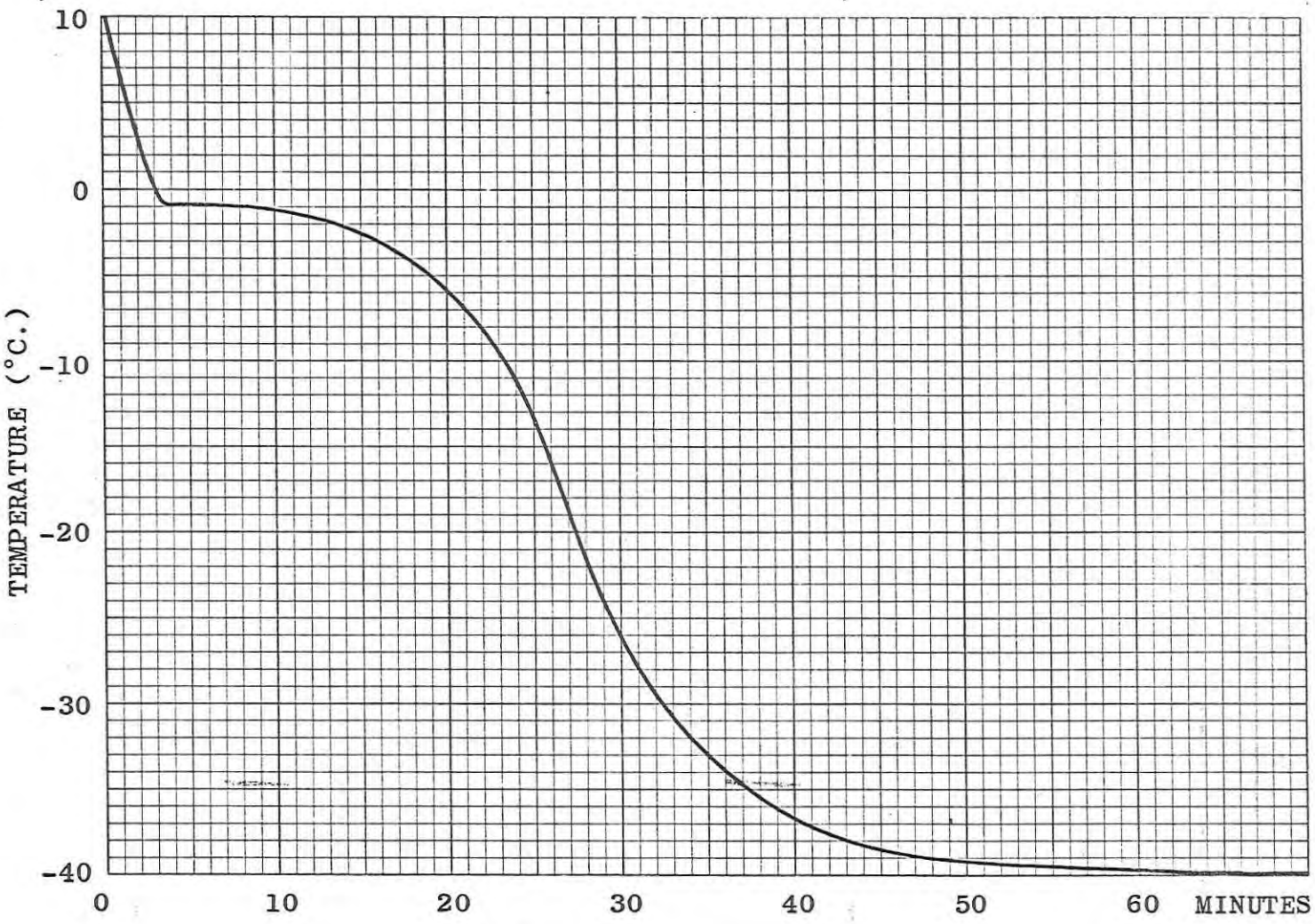


FIGURE 18. FREEZING OF PEACH SLICES IN STILL AIR AT -40°C.

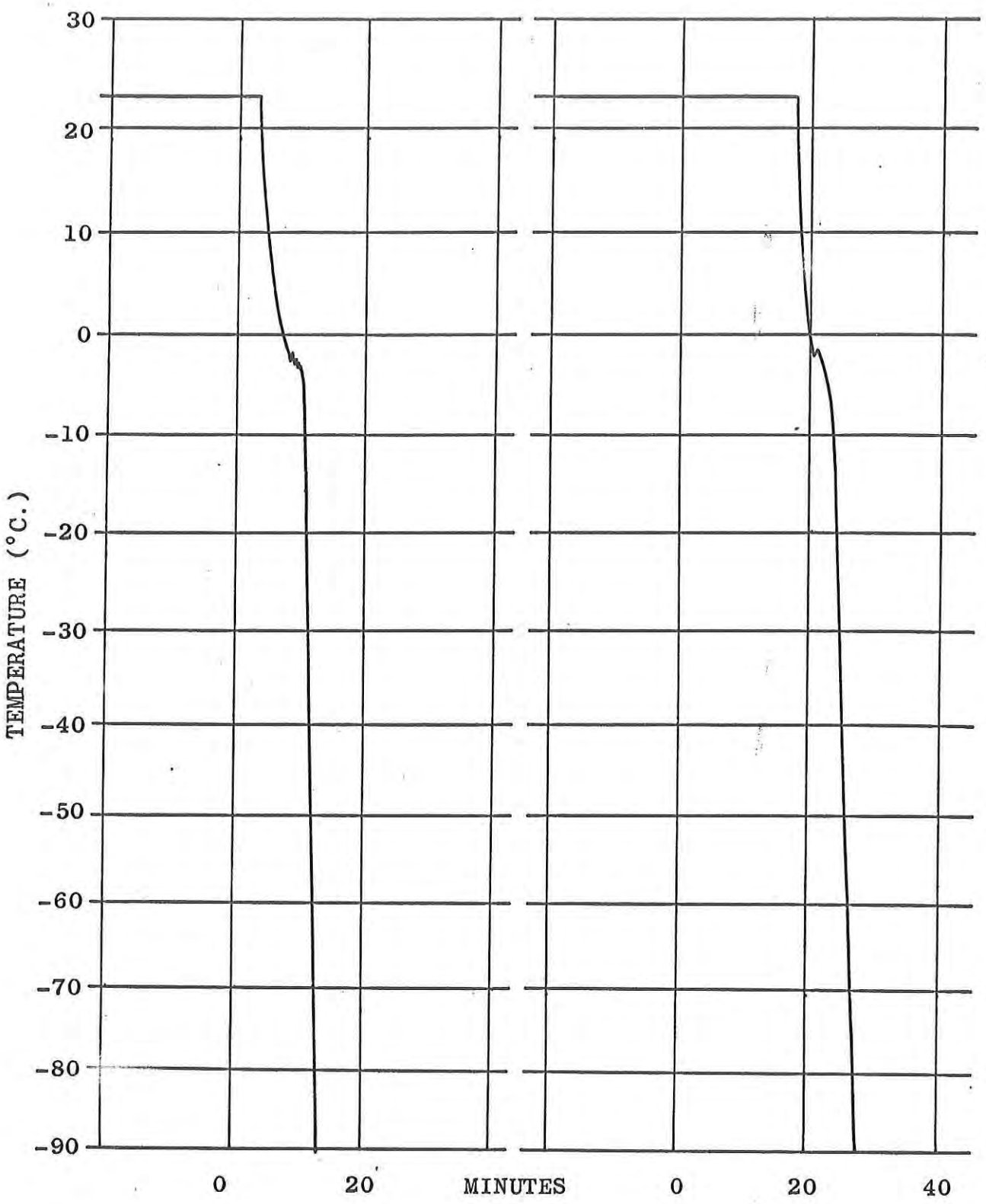


FIGURE 19. RAPID FREEZING OF PEACH SLICES.

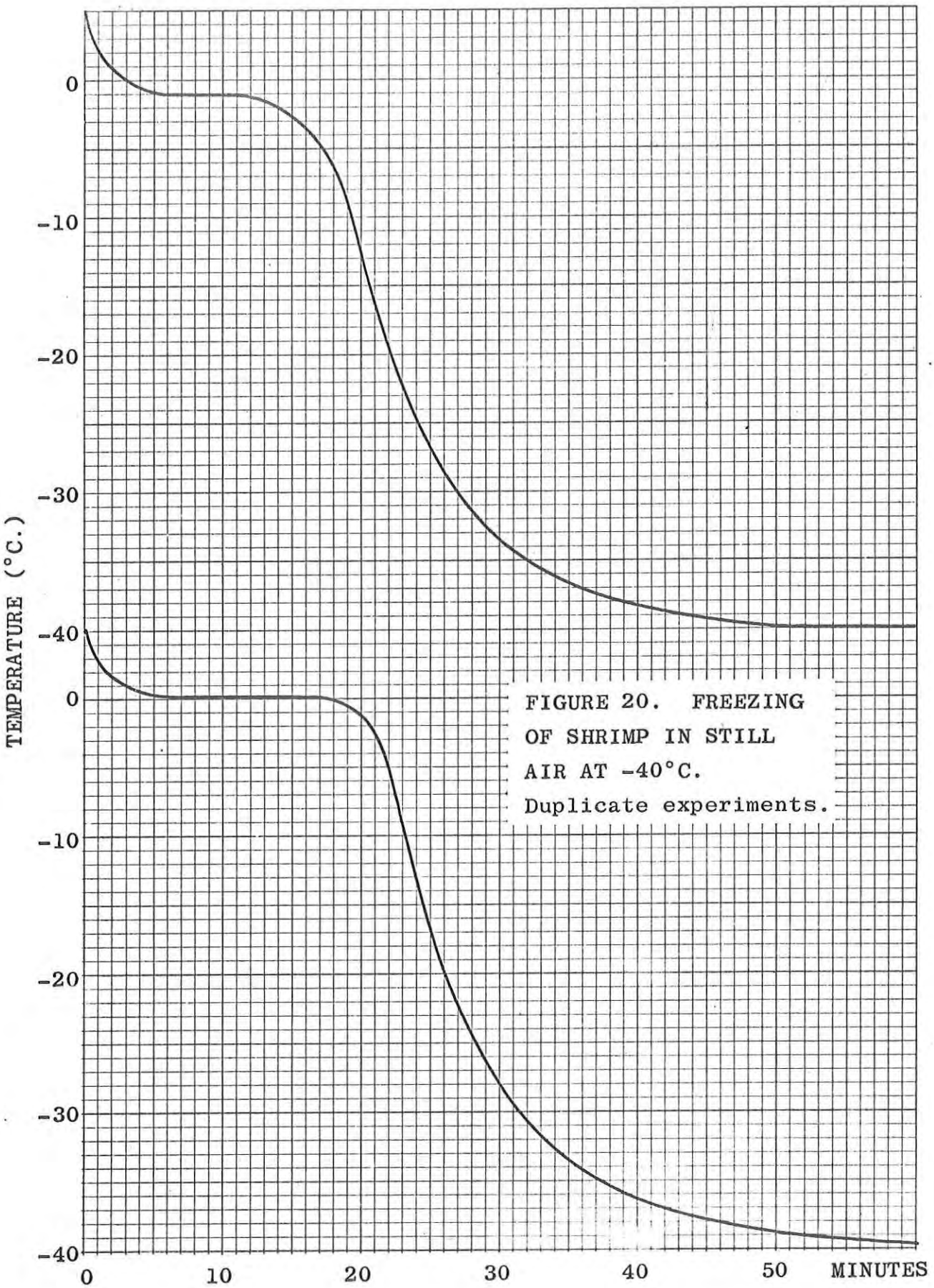


FIGURE 20. FREEZING OF SHRIMP IN STILL AIR AT -40°C. Duplicate experiments.

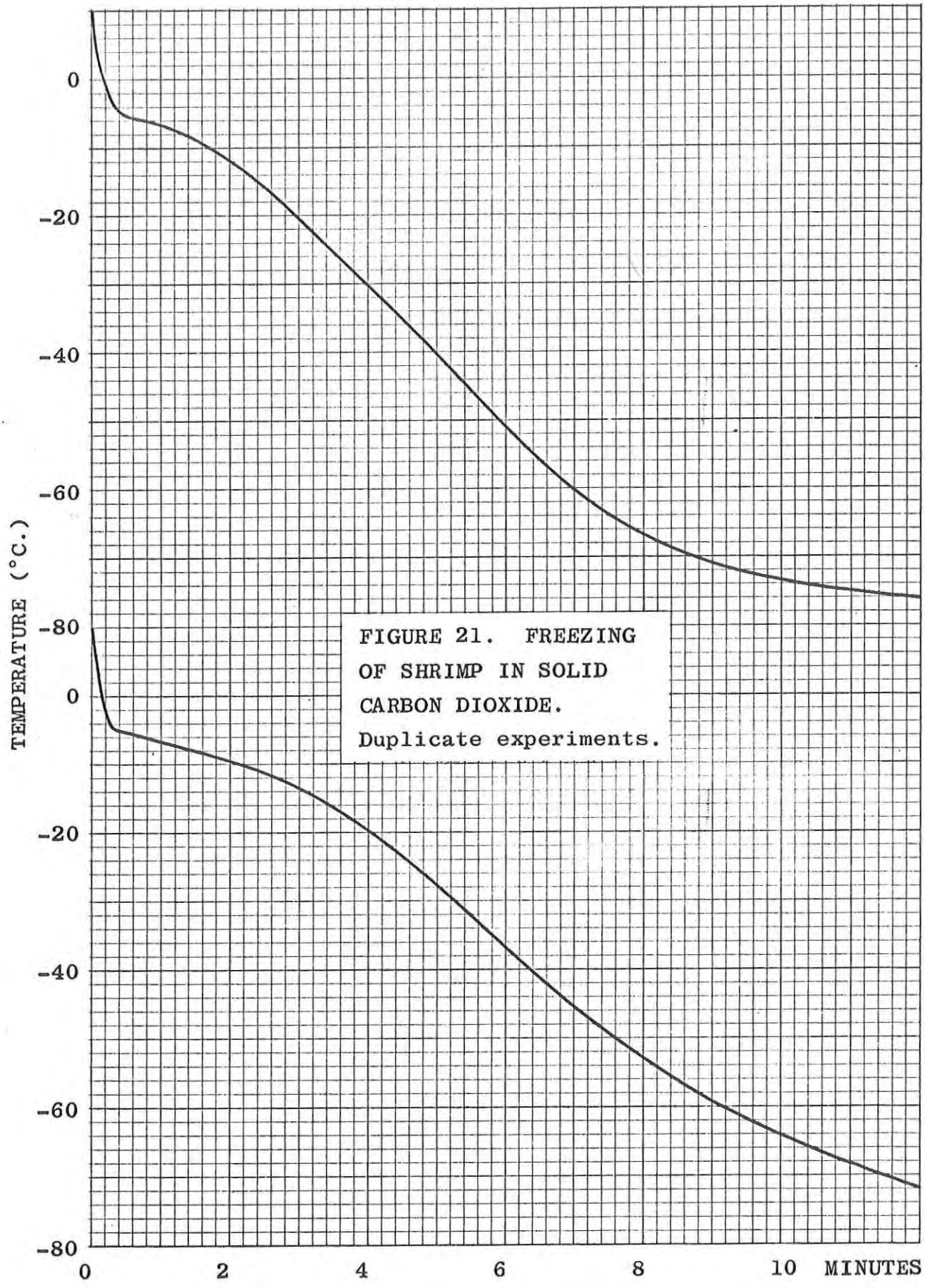


FIGURE 21. FREEZING OF SHRIMP IN SOLID CARBON DIOXIDE. Duplicate experiments.

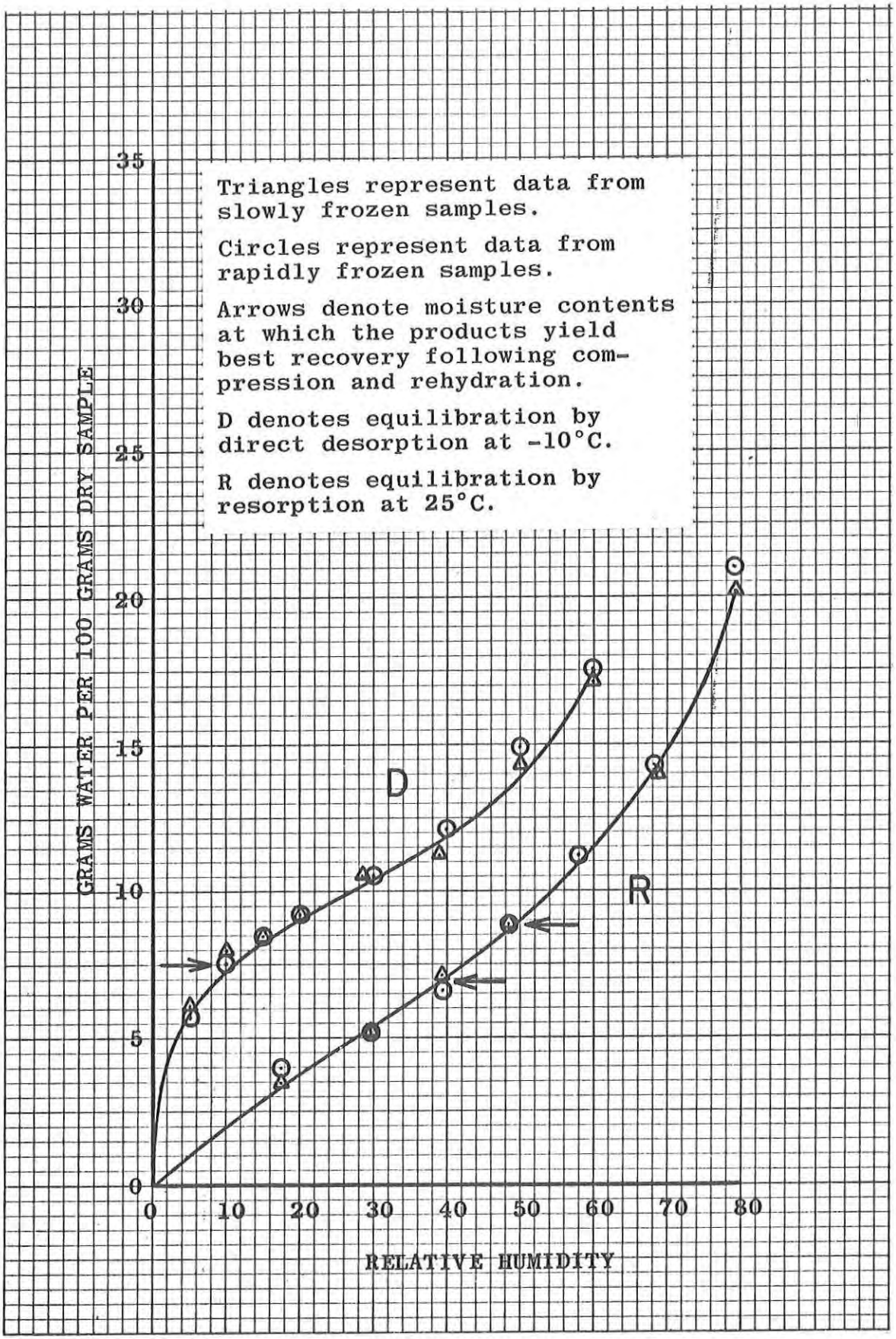


FIGURE 22. SORPTION ISOTHERMS. FREEZE-DRIED RAW BEEF

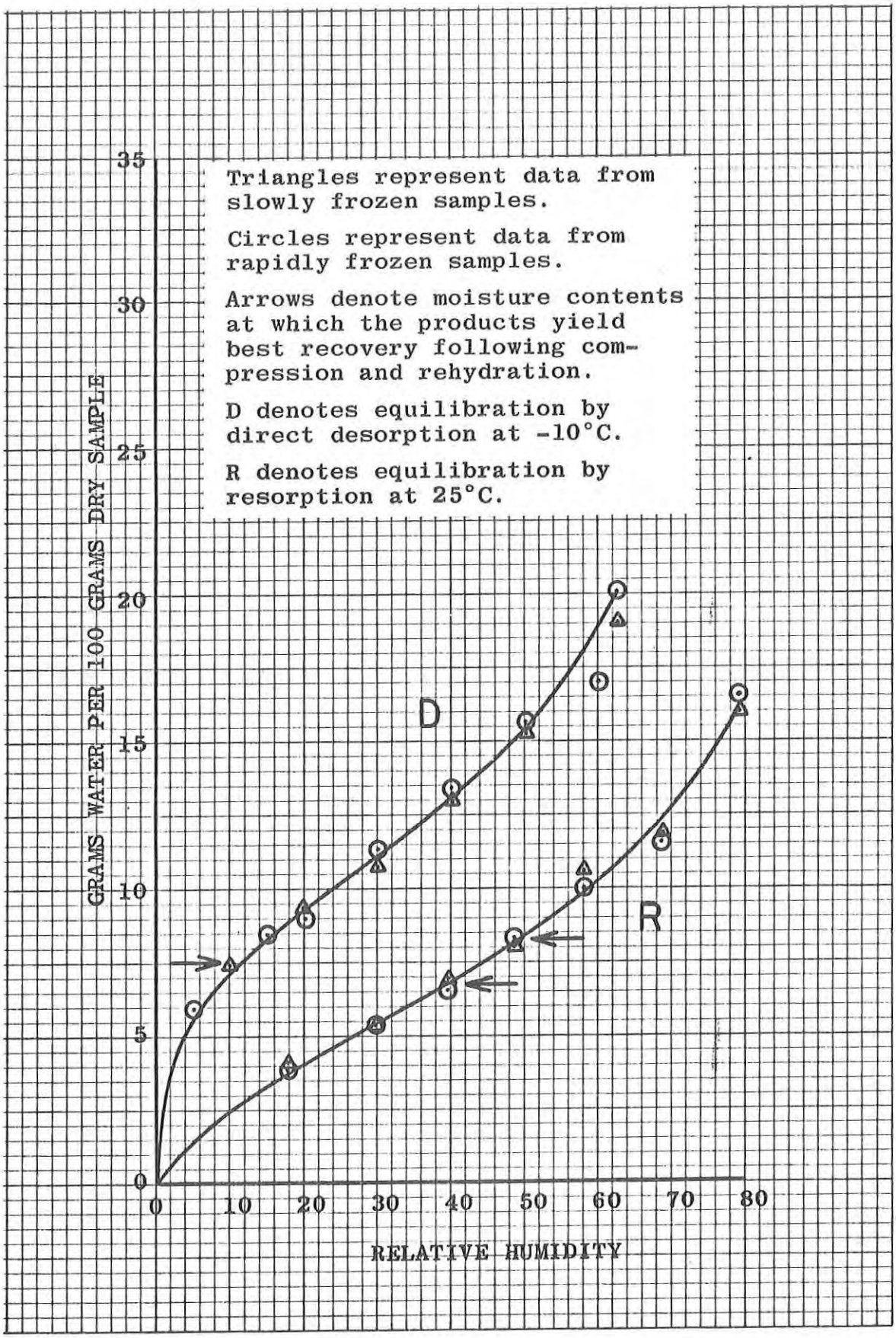


FIGURE 23. SORPTION ISOTHERMS. FREEZE-DRIED COOKED BEEF

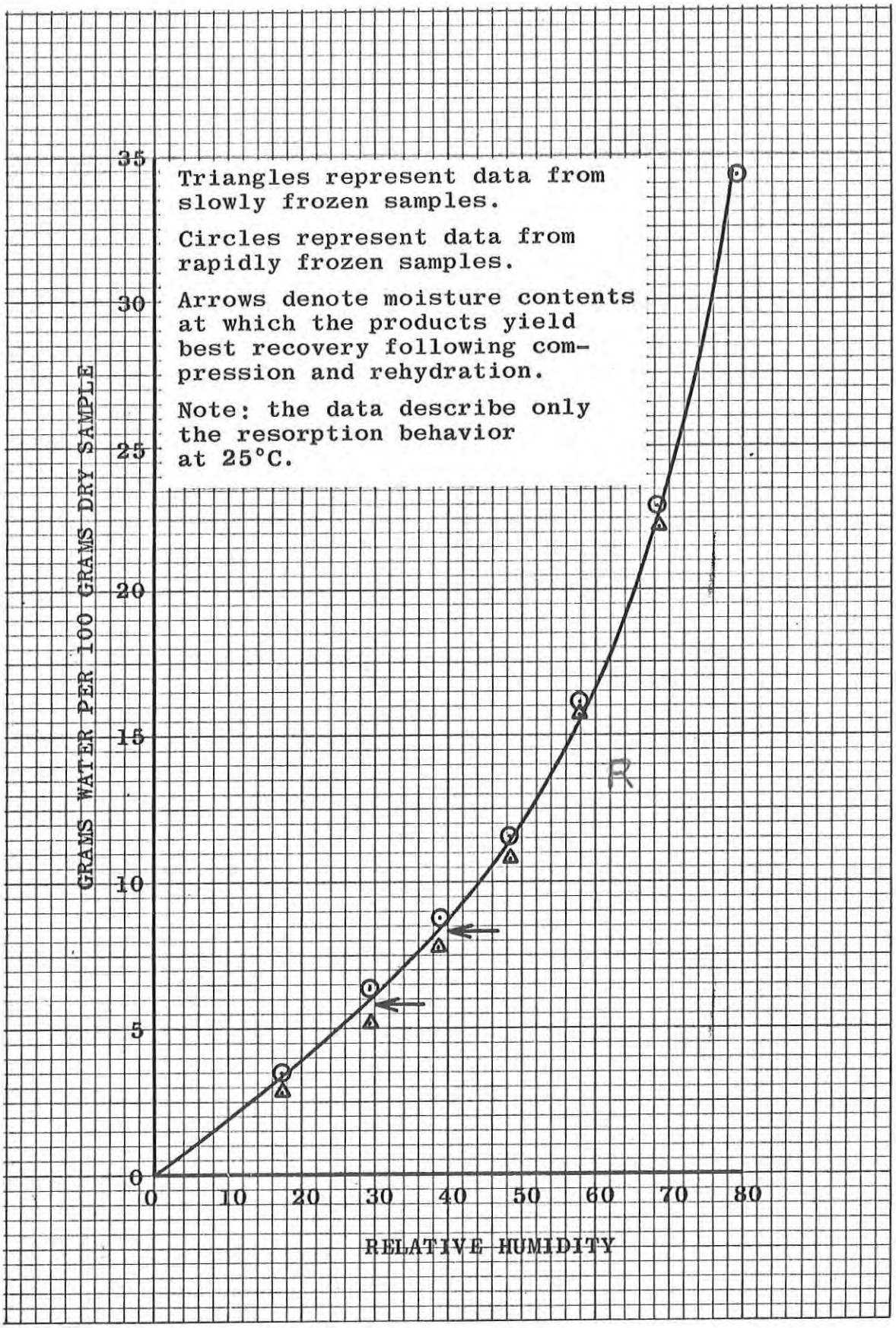


FIGURE 24. SORPTION ISOTHERMS. FREEZE-DRIED RAW CABBAGE

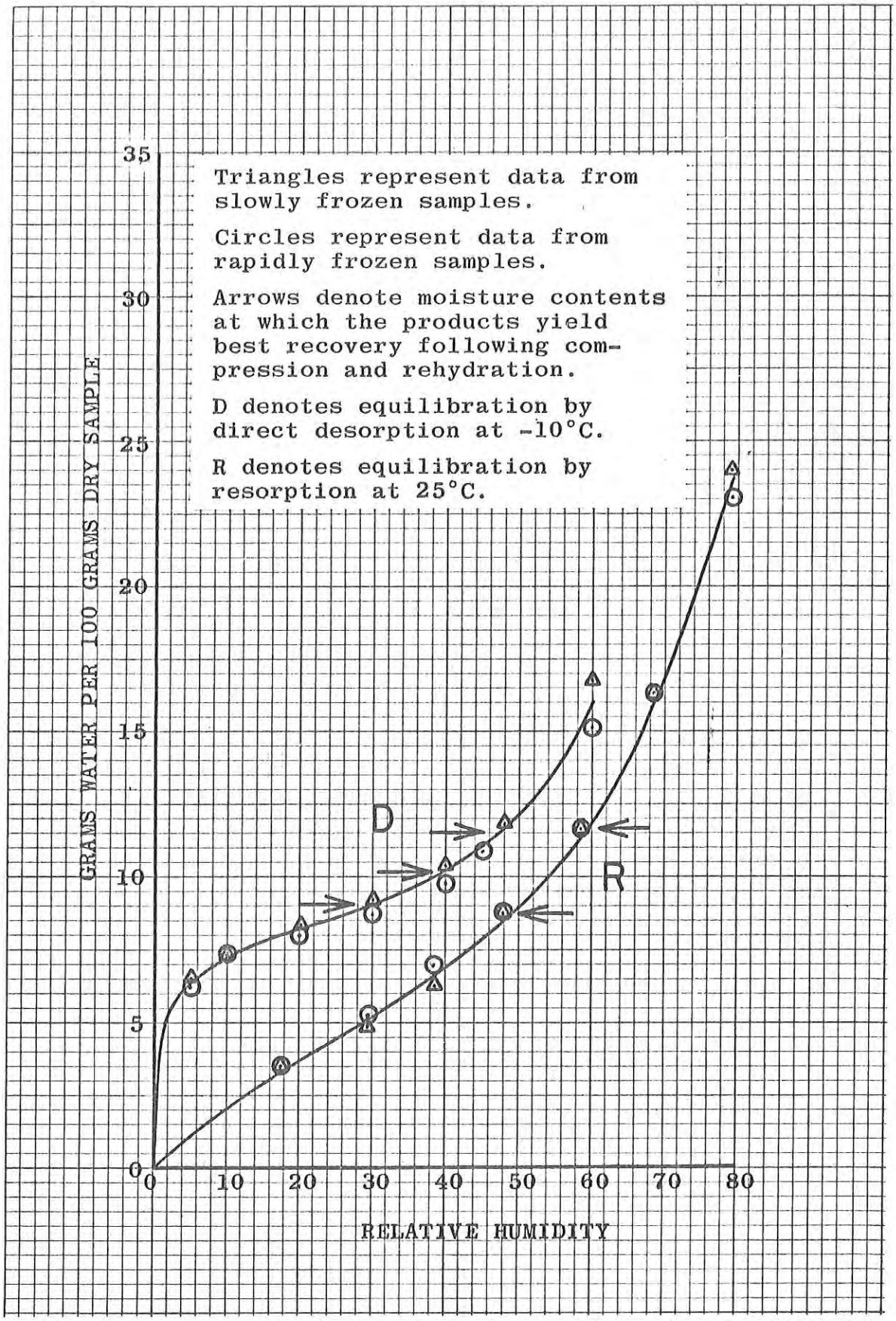


FIGURE 25. SORPTION ISOTHERMS. FREEZE-DRIED COOKED CARROTS

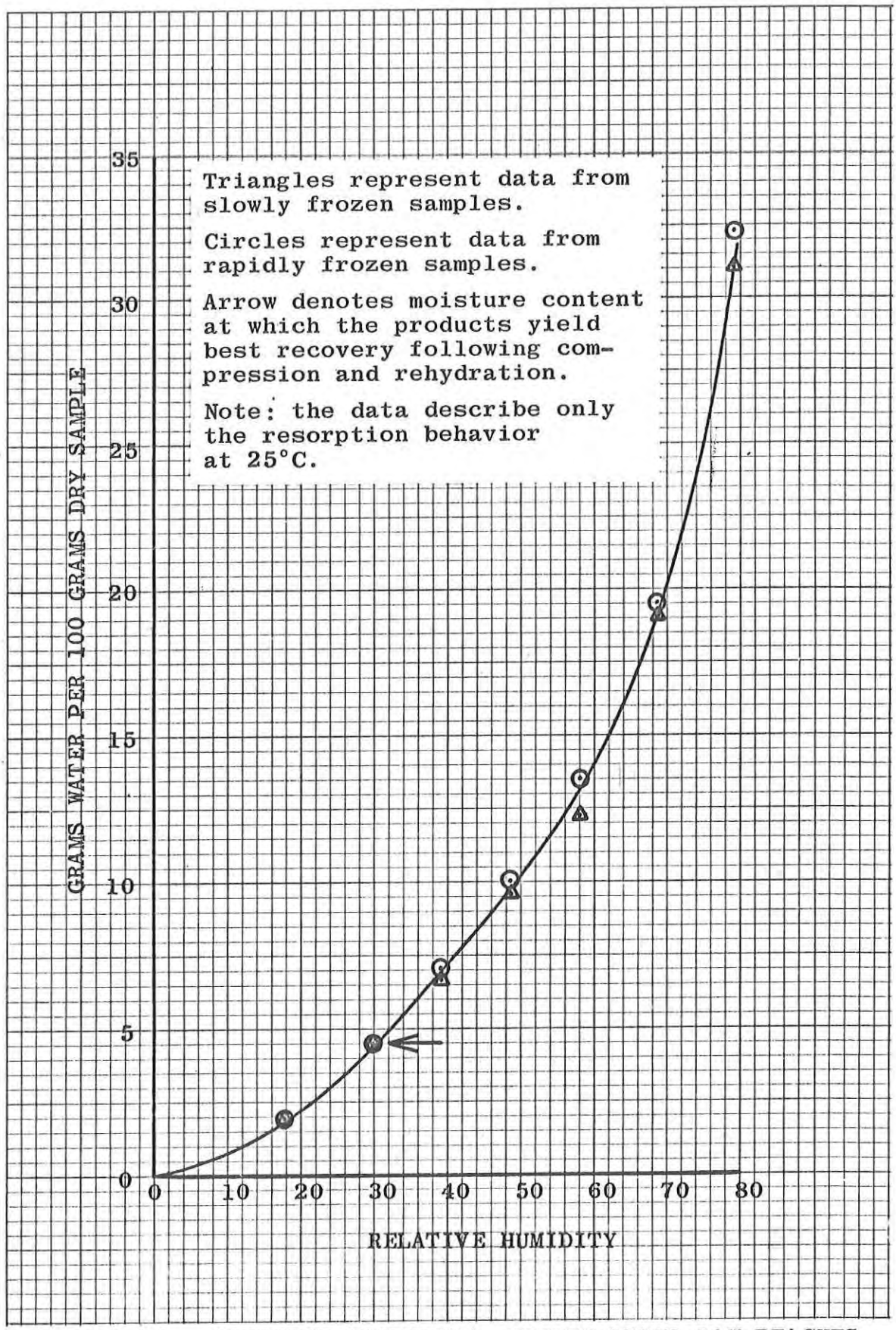


FIGURE 26. SORPTION ISOTHERMS. FREEZE-DRIED RAW PEACHES

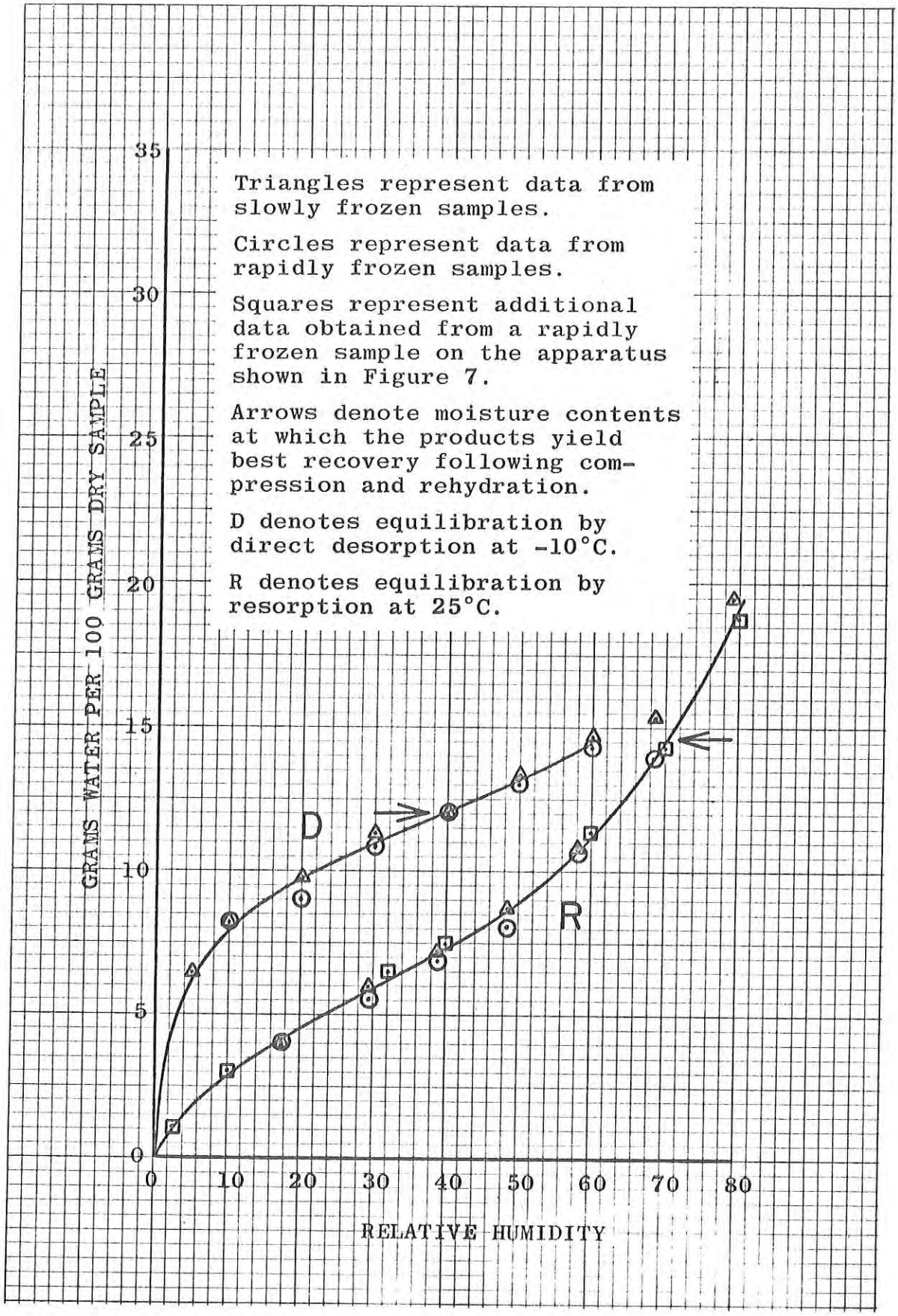


FIGURE 27. SORPTION ISOTHERMS. FREEZE-DRIED COOKED PEAS

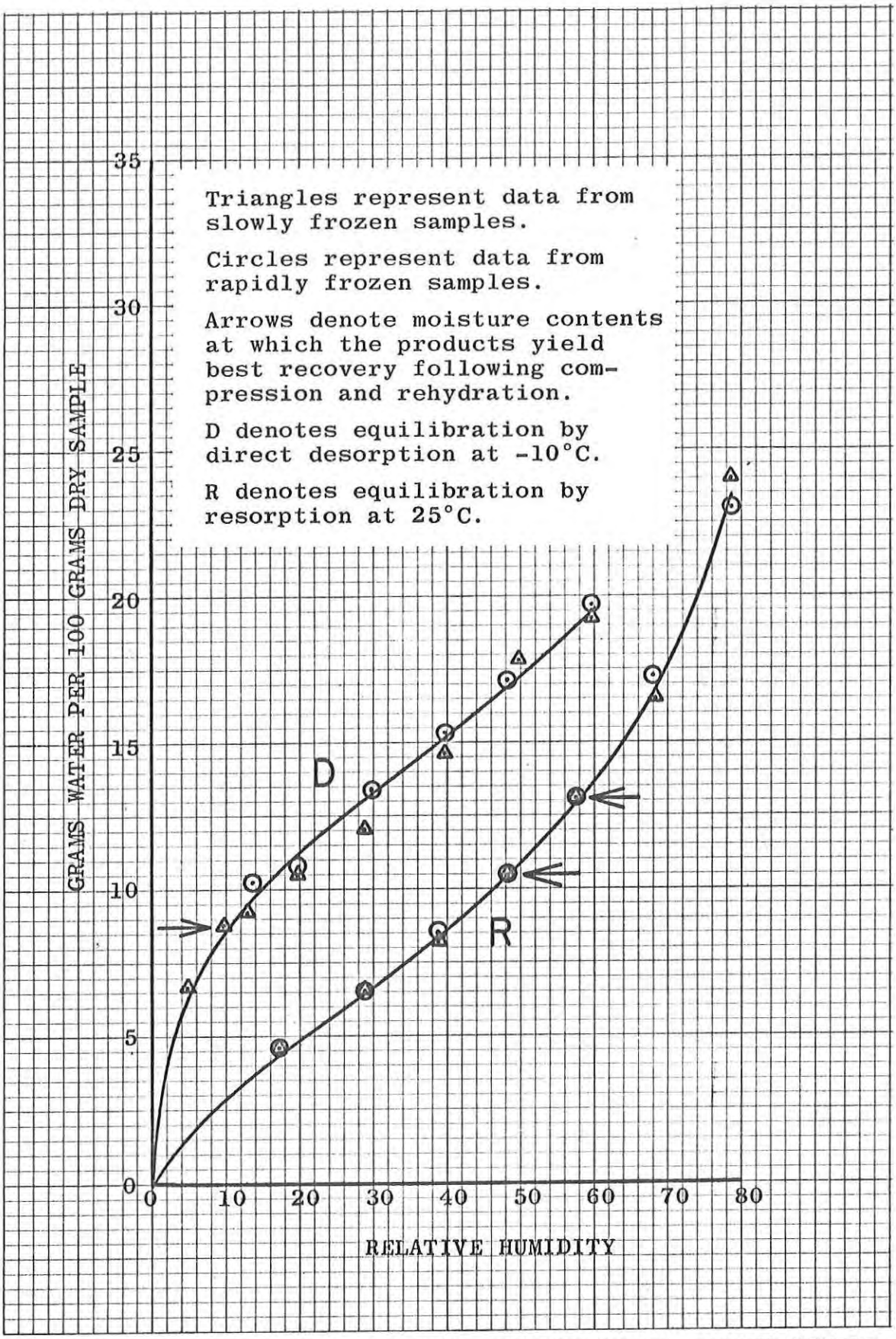


FIGURE 28. SORPTION ISOTHERMS. FREEZE-DRIED COOKED SHRIMP

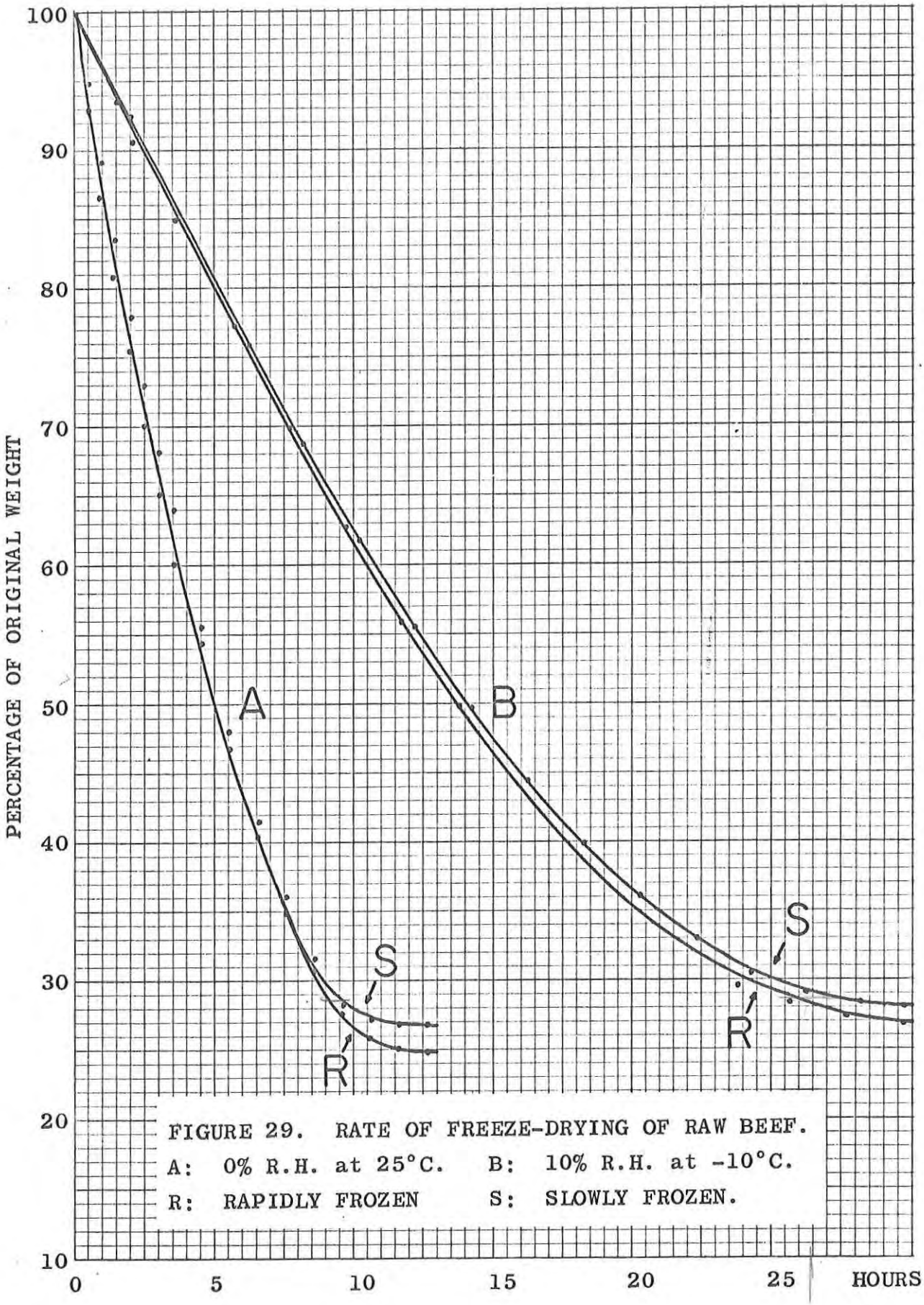


FIGURE 29. RATE OF FREEZE-DRYING OF RAW BEEF.
A: 0% R.H. at 25°C. B: 10% R.H. at -10°C.
R: RAPIDLY FROZEN S: SLOWLY FROZEN.

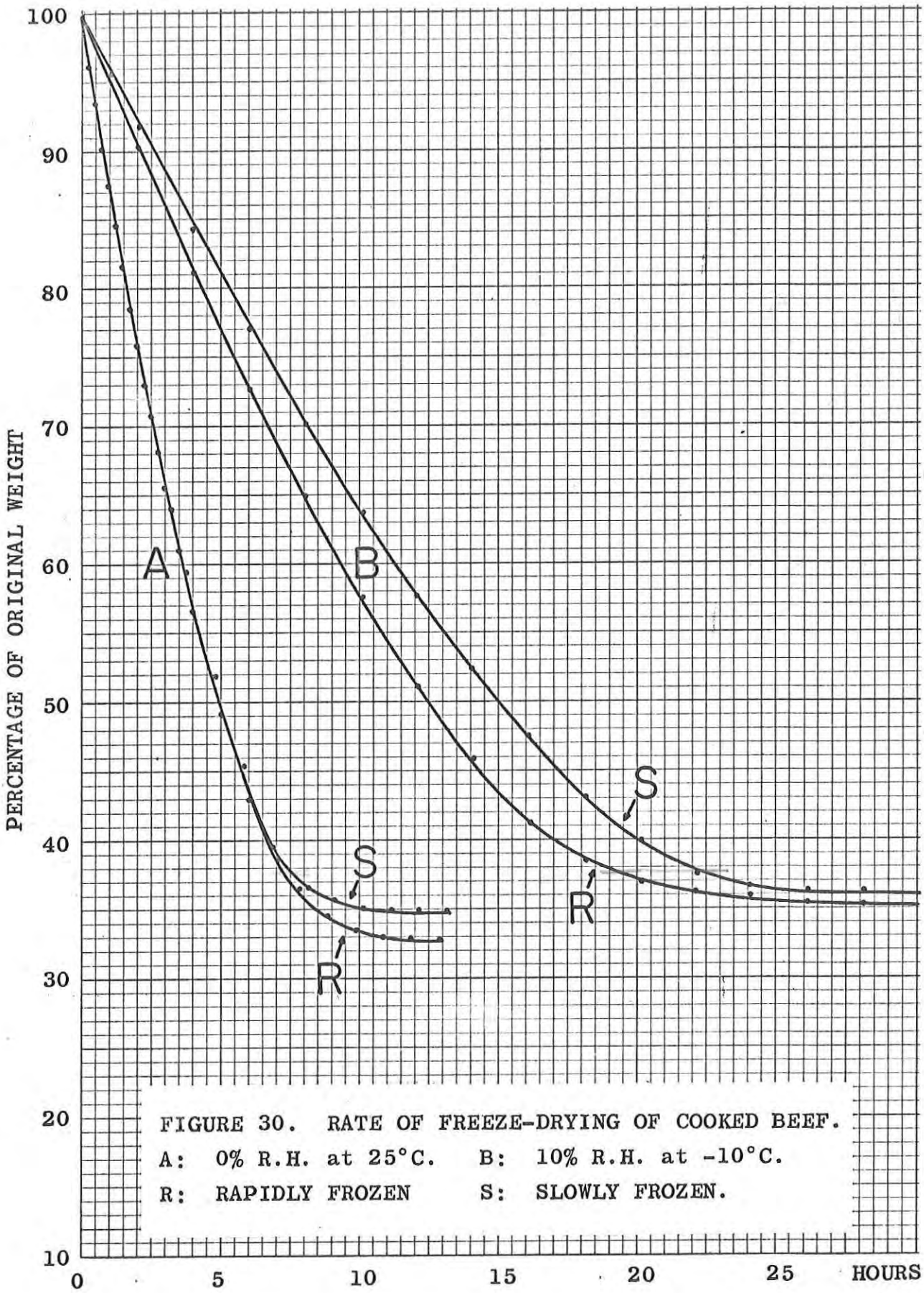


FIGURE 30. RATE OF FREEZE-DRYING OF COOKED BEEF.
A: 0% R.H. at 25°C. B: 10% R.H. at -10°C.
R: RAPIDLY FROZEN S: SLOWLY FROZEN.

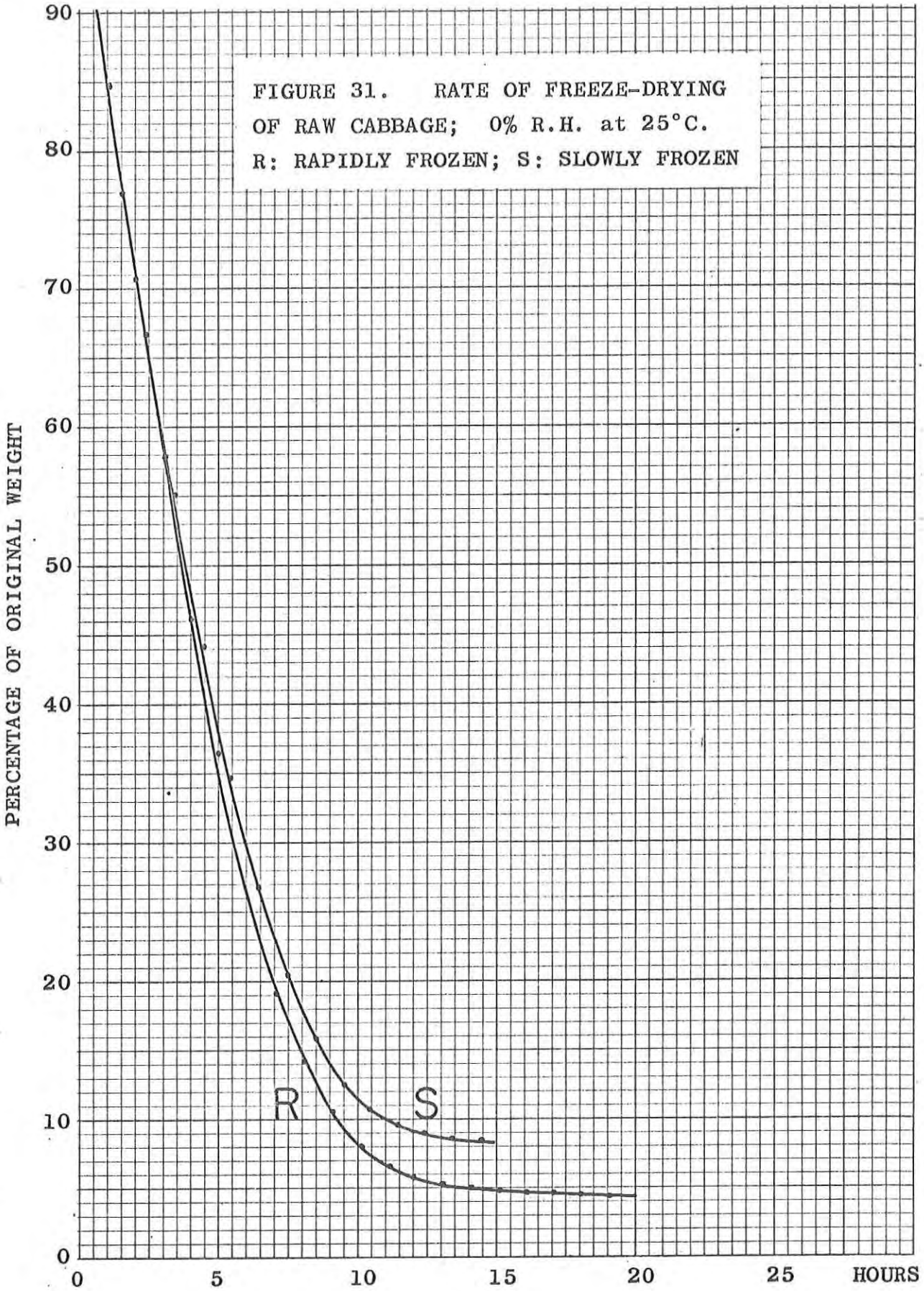


FIGURE 31. RATE OF FREEZE-DRYING OF RAW CABBAGE; 0% R.H. at 25°C.
R: RAPIDLY FROZEN; S: SLOWLY FROZEN
PERCENTAGE OF ORIGINAL WEIGHT
90
80
70
60
50
40
30
20
10
0
0
5
10
15
20
25
HOURS
R
S

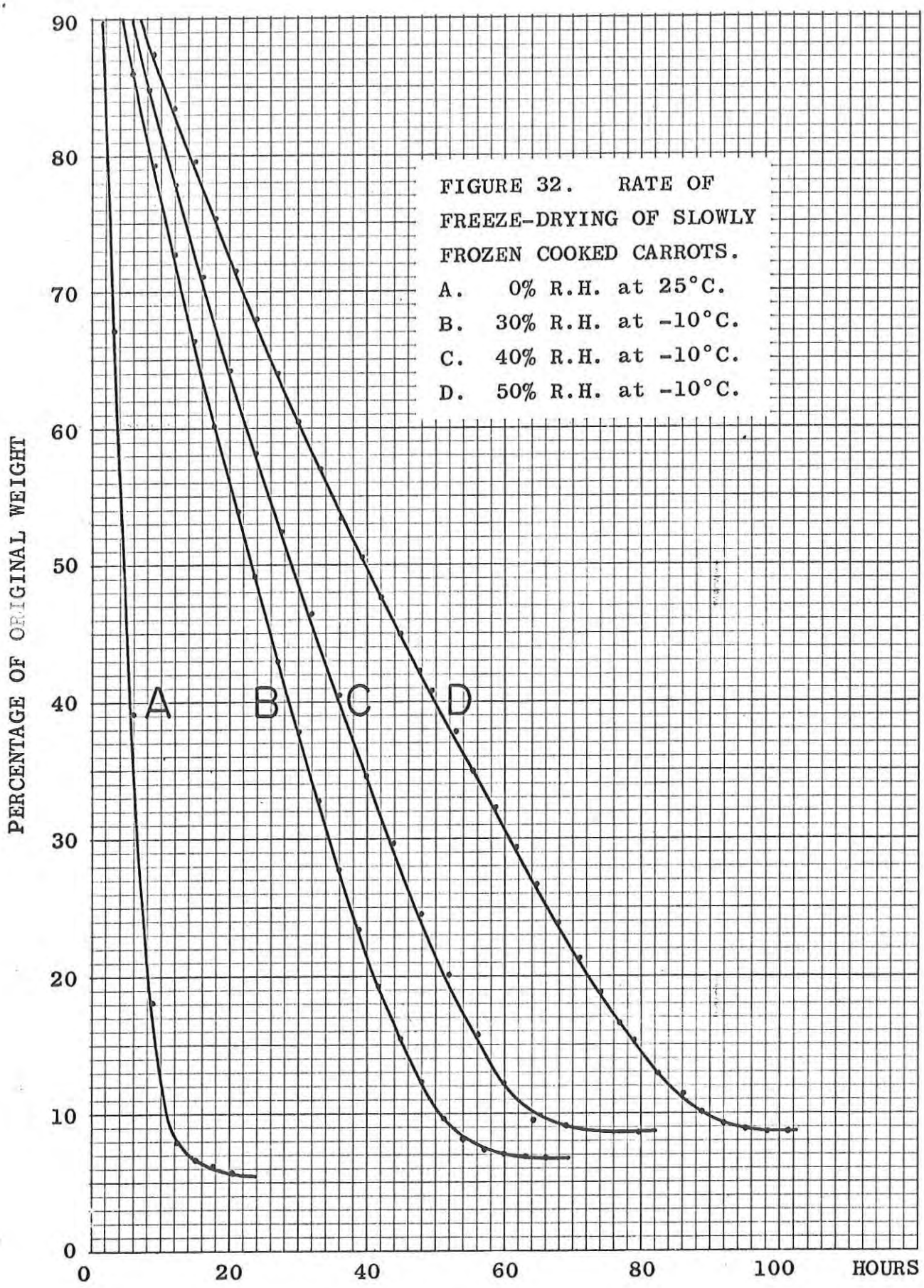


FIGURE 32. RATE OF FREEZE-DRYING OF SLOWLY FROZEN COOKED CARROTS.
A. 0% R.H. at 25°C.
B. 30% R.H. at -10°C.
C. 40% R.H. at -10°C.
D. 50% R.H. at -10°C.
PERCENTAGE OF ORIGINAL WEIGHT
90
80
70
60
50
40
30
20
10
0
A
B
C
D
0
20
40
60
80
100
HOURS

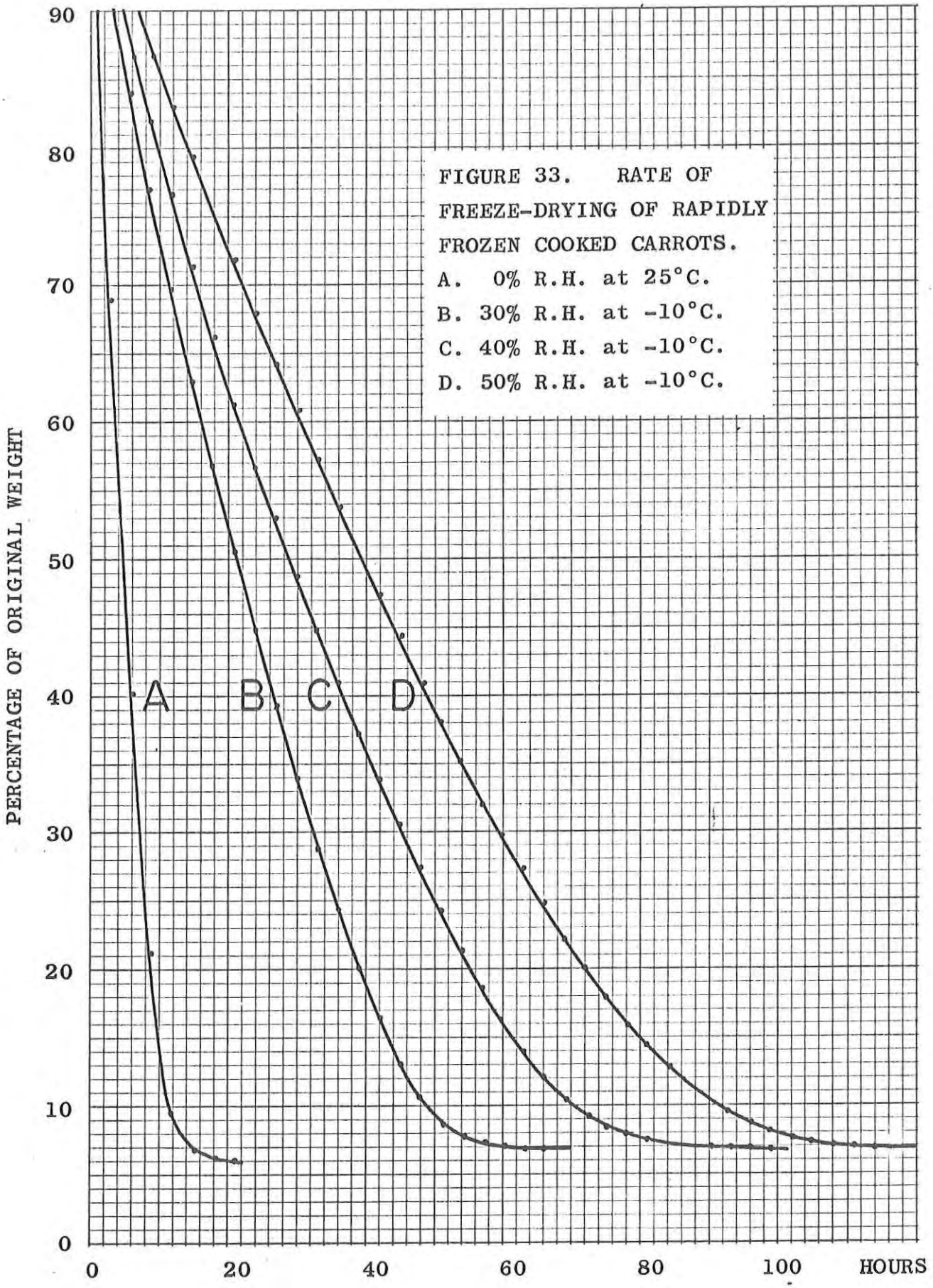


FIGURE 33. RATE OF FREEZE-DRYING OF RAPIDLY FROZEN COOKED CARROTS.

A. 0% R.H. at 25°C.

B. 30% R.H. at -10°C.

C. 40% R.H. at -10°C.

D. 50% R.H. at -10°C.

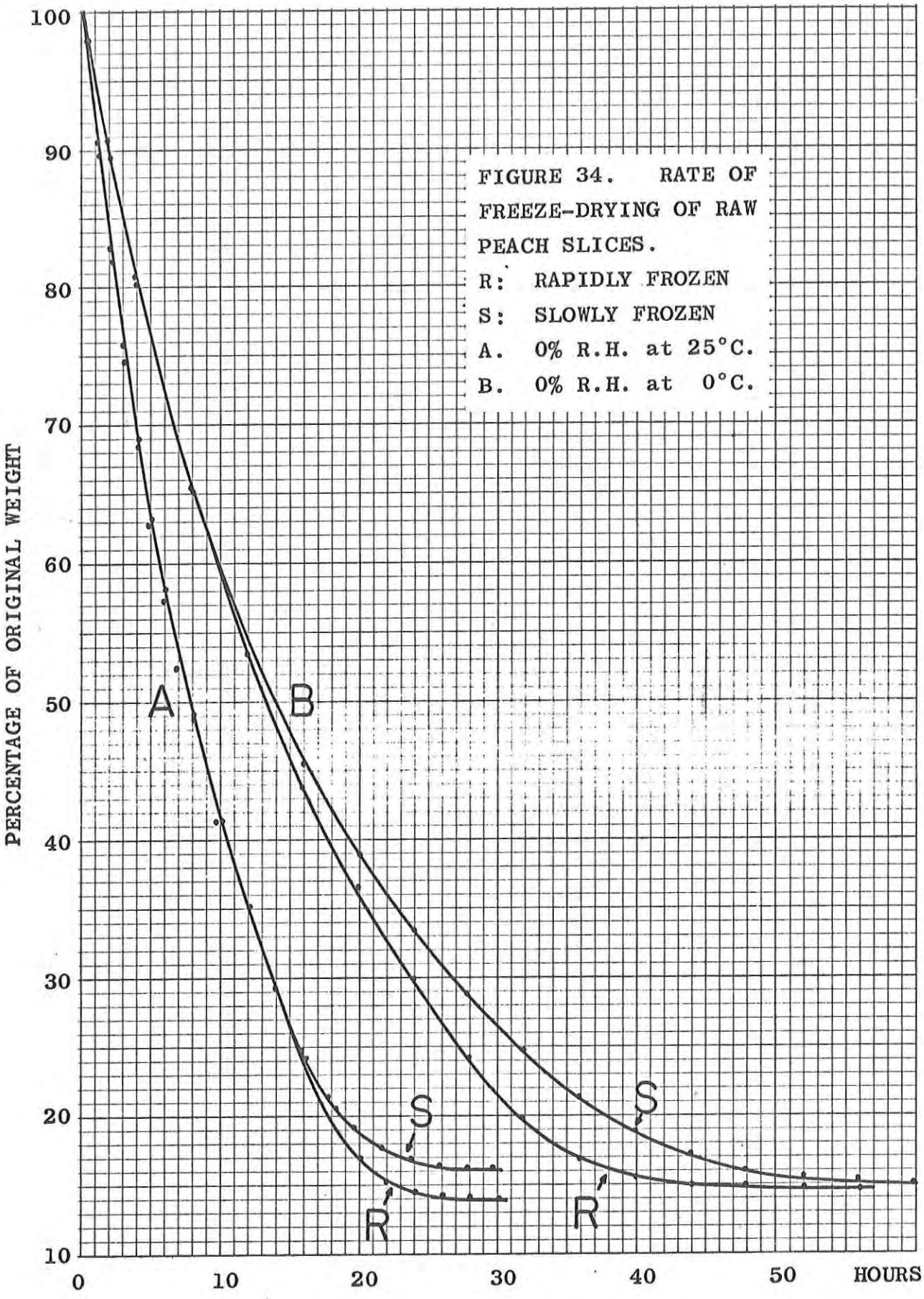


FIGURE 34. RATE OF FREEZE-DRYING OF RAW PEACH SLICES.
R: RAPIDLY FROZEN
S: SLOWLY FROZEN
A. 0% R.H. at 25°C.
B. 0% R.H. at 0°C.
PERCENTAGE OF ORIGINAL WEIGHT
100
90
80
70
60
50
40
30
20
10
0
10
20
30
40
50
HOURS
A
B
S
S
R
R

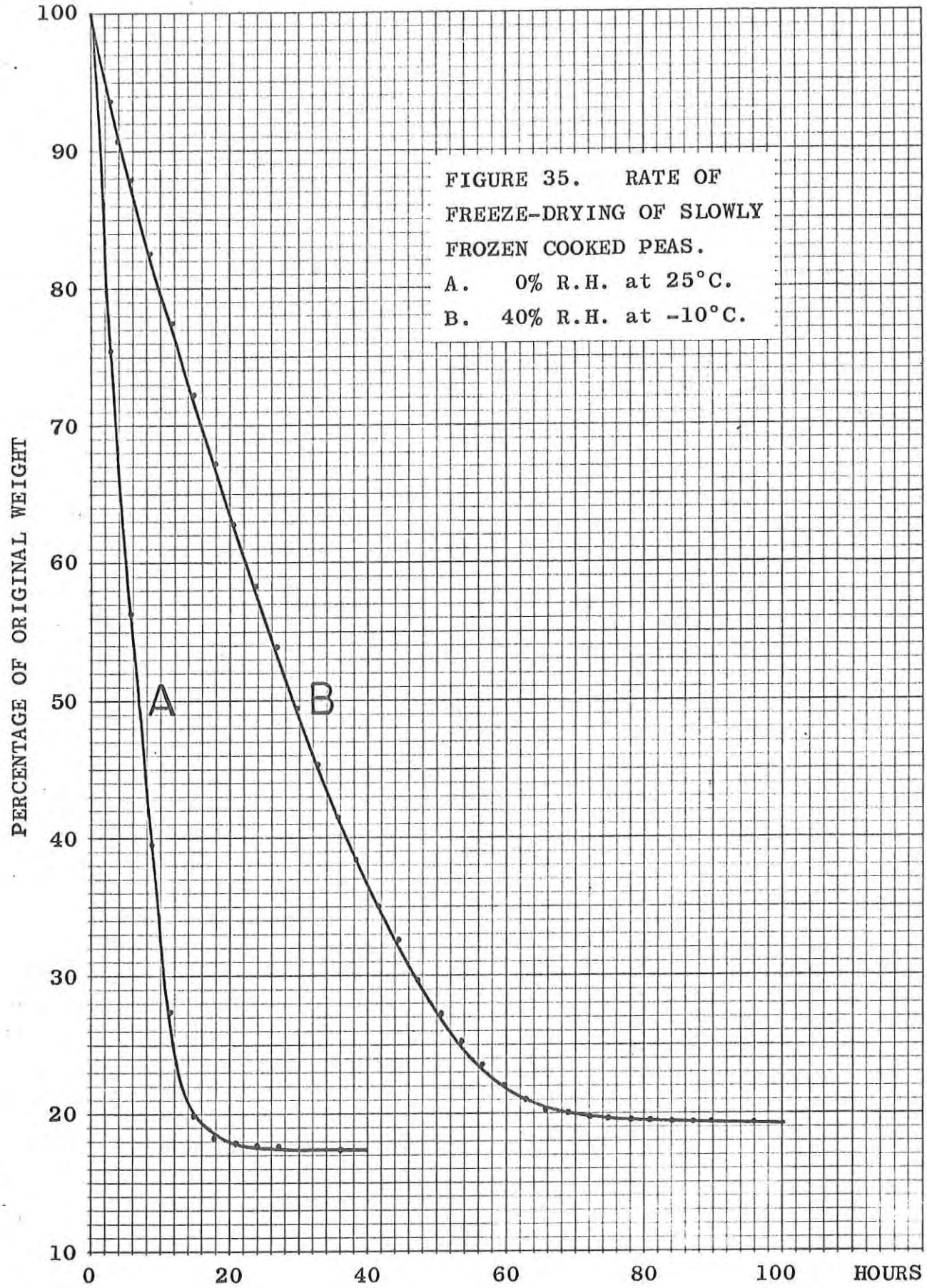


FIGURE 35. RATE OF FREEZE-DRYING OF SLOWLY FROZEN COOKED PEAS.
A. 0% R.H. at 25°C.
B. 40% R.H. at -10°C.

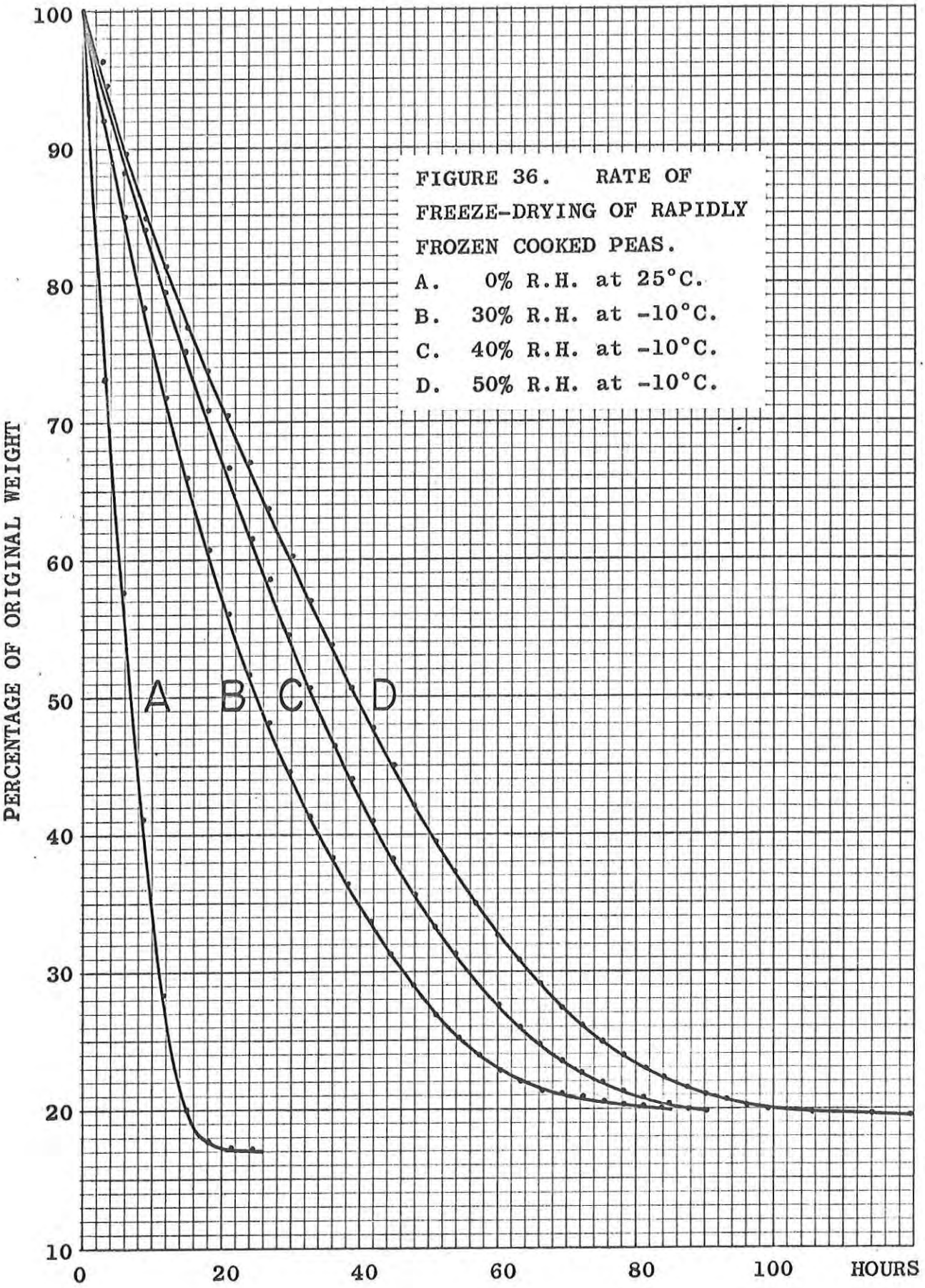


FIGURE 36. RATE OF FREEZE-DRYING OF RAPIDLY FROZEN COOKED PEAS.

A. 0% R.H. at 25°C.
B. 30% R.H. at -10°C.
C. 40% R.H. at -10°C.
D. 50% R.H. at -10°C.

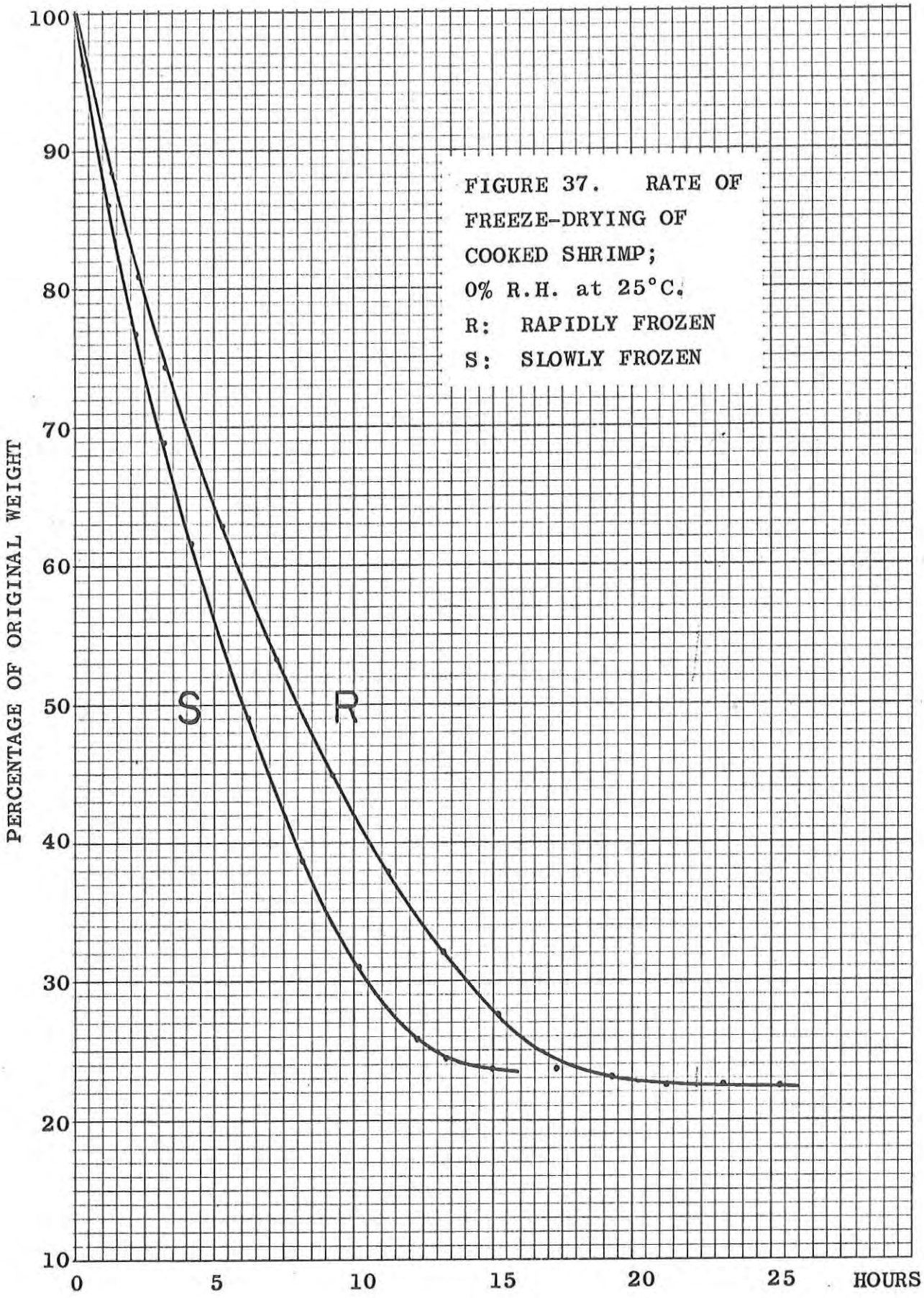


FIGURE 37. RATE OF FREEZE-DRYING OF COOKED SHRIMP; 0% R.H. at 25°C.
R: RAPIDLY FROZEN
S: SLOWLY FROZEN

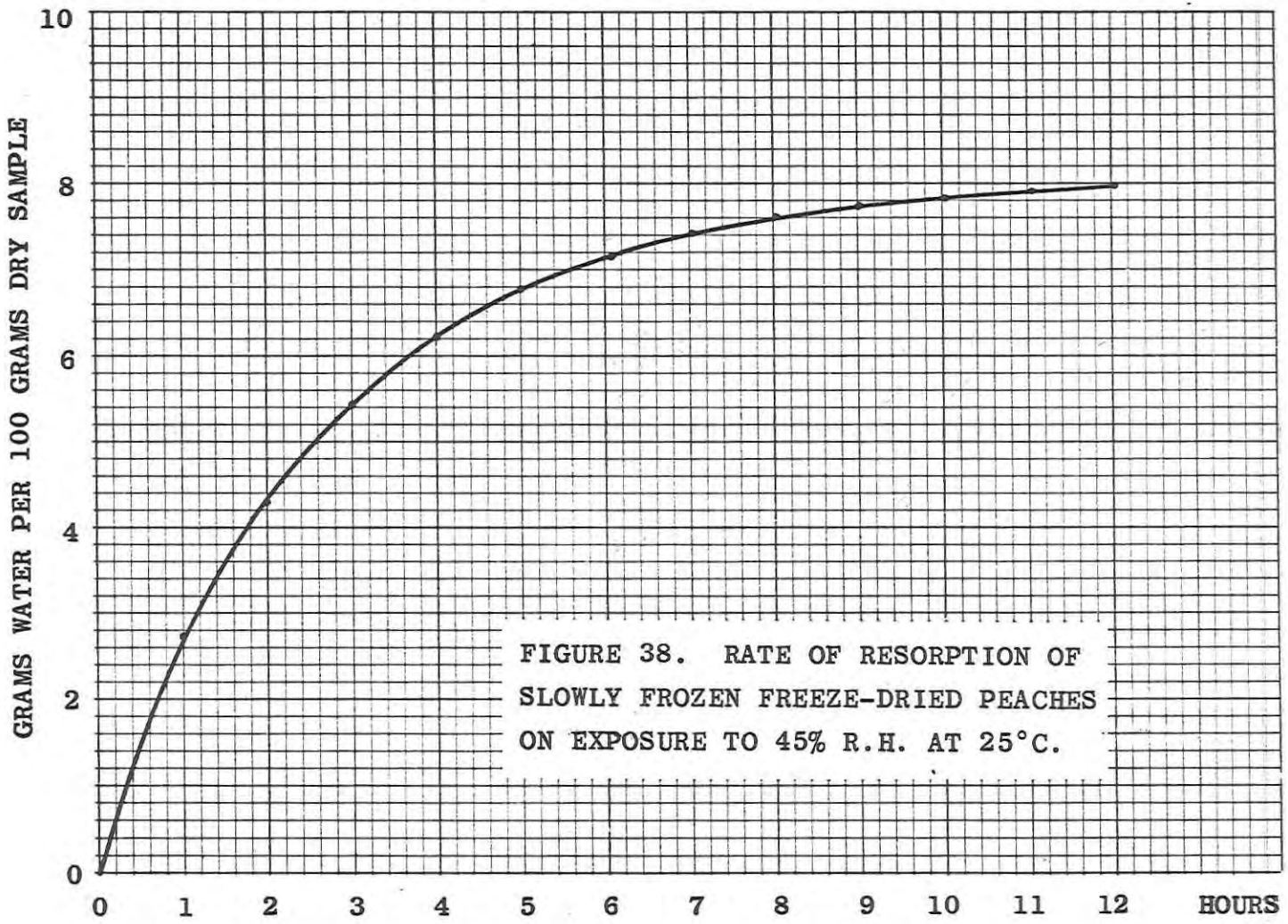


FIGURE 38. RATE OF RESORPTION OF SLOWLY FROZEN FREEZE-DRIED PEACHES ON EXPOSURE TO 45% R.H. AT 25°C.

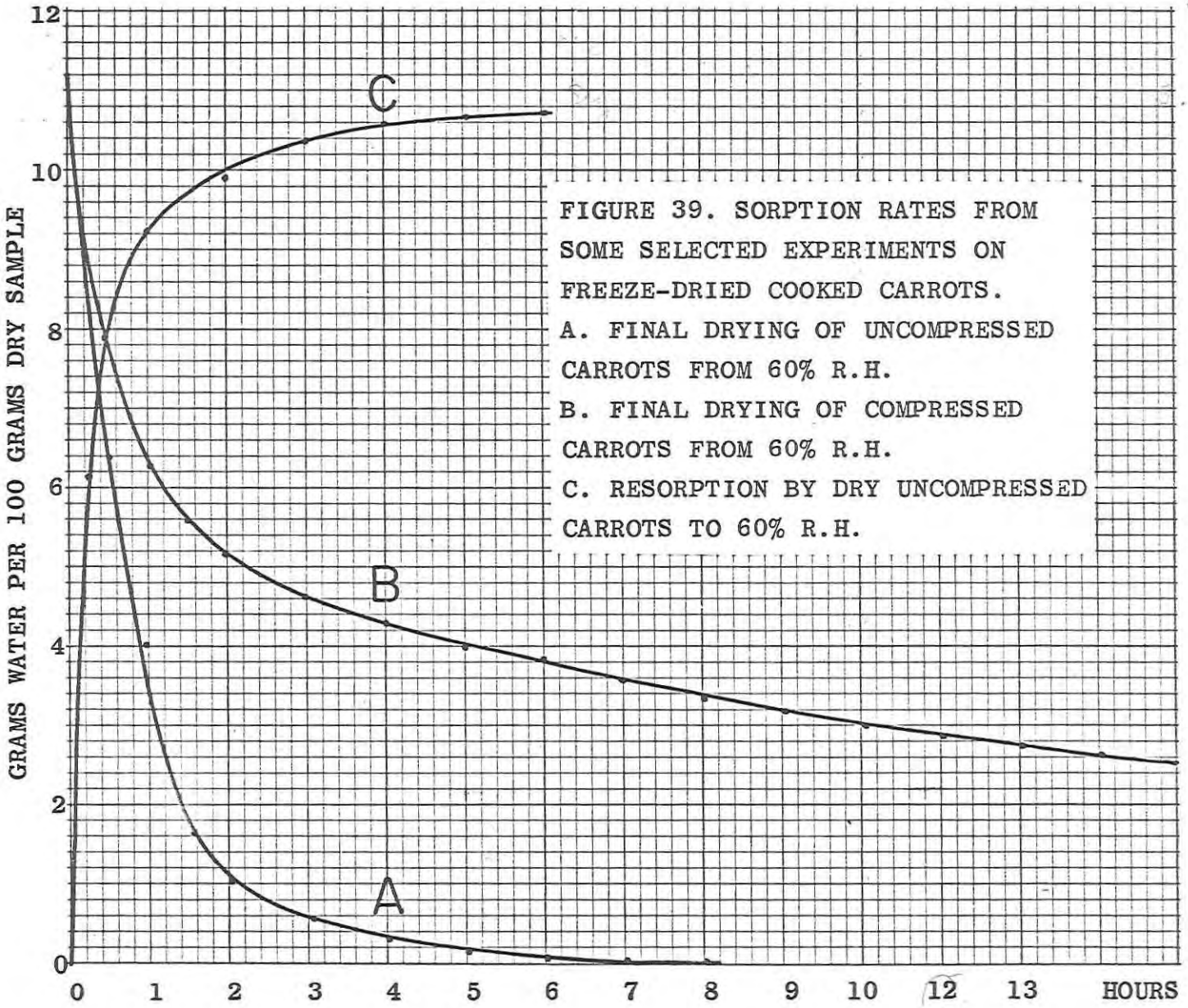


FIGURE 39. SORPTION RATES FROM SOME SELECTED EXPERIMENTS ON FREEZE-DRIED COOKED CARROTS.
A. FINAL DRYING OF UNCOMPRESSED CARROTS FROM 60% R.H.
B. FINAL DRYING OF COMPRESSED CARROTS FROM 60% R.H.
C. RESORPTION BY DRY UNCOMPRESSED CARROTS TO 60% R.H.

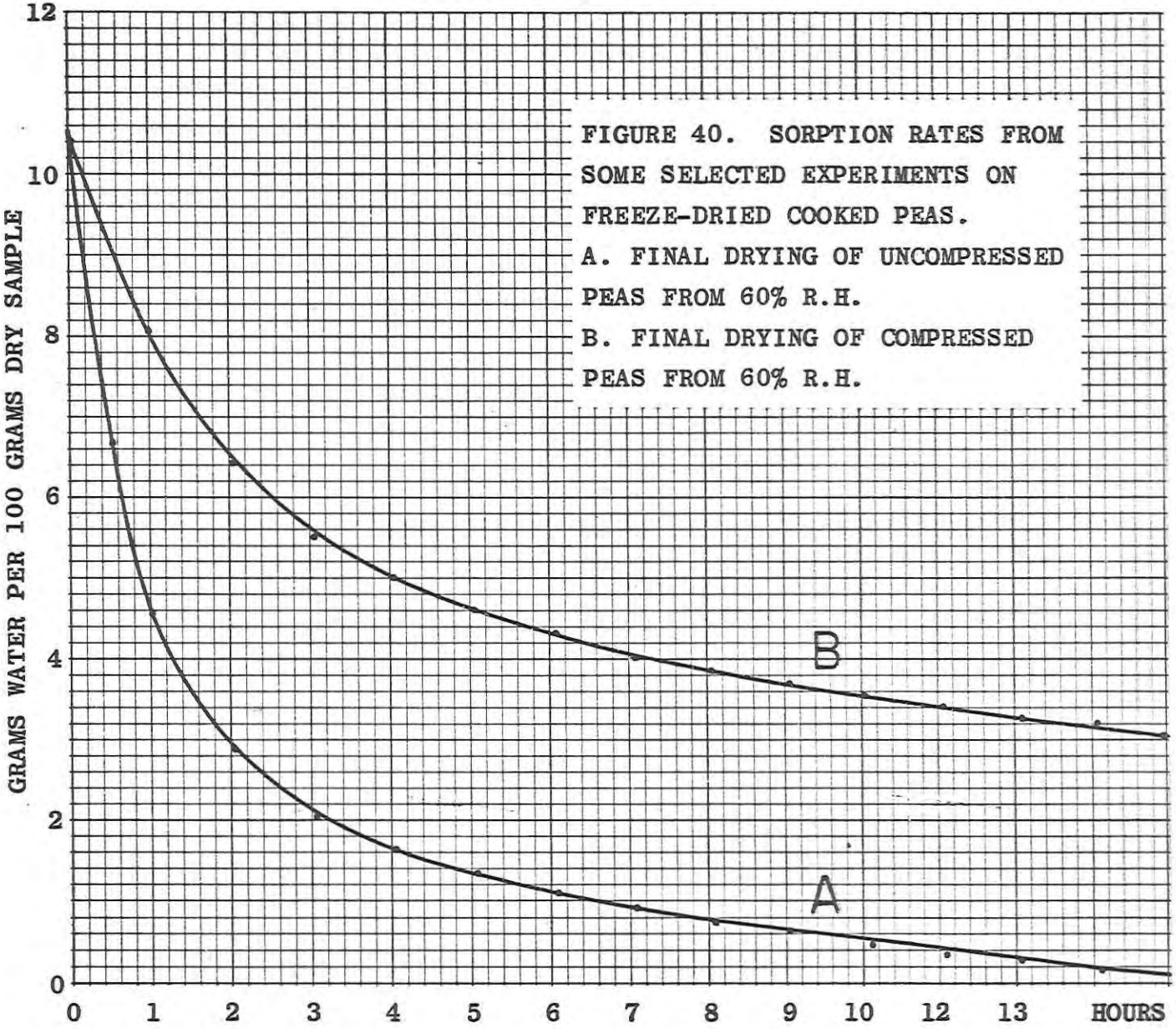


FIGURE 40. SORPTION RATES FROM SOME SELECTED EXPERIMENTS ON FREEZE-DRIED COOKED PEAS.
A. FINAL DRYING OF UNCOMPRESSED PEAS FROM 60% R.H.
B. FINAL DRYING OF COMPRESSED PEAS FROM 60% R.H.

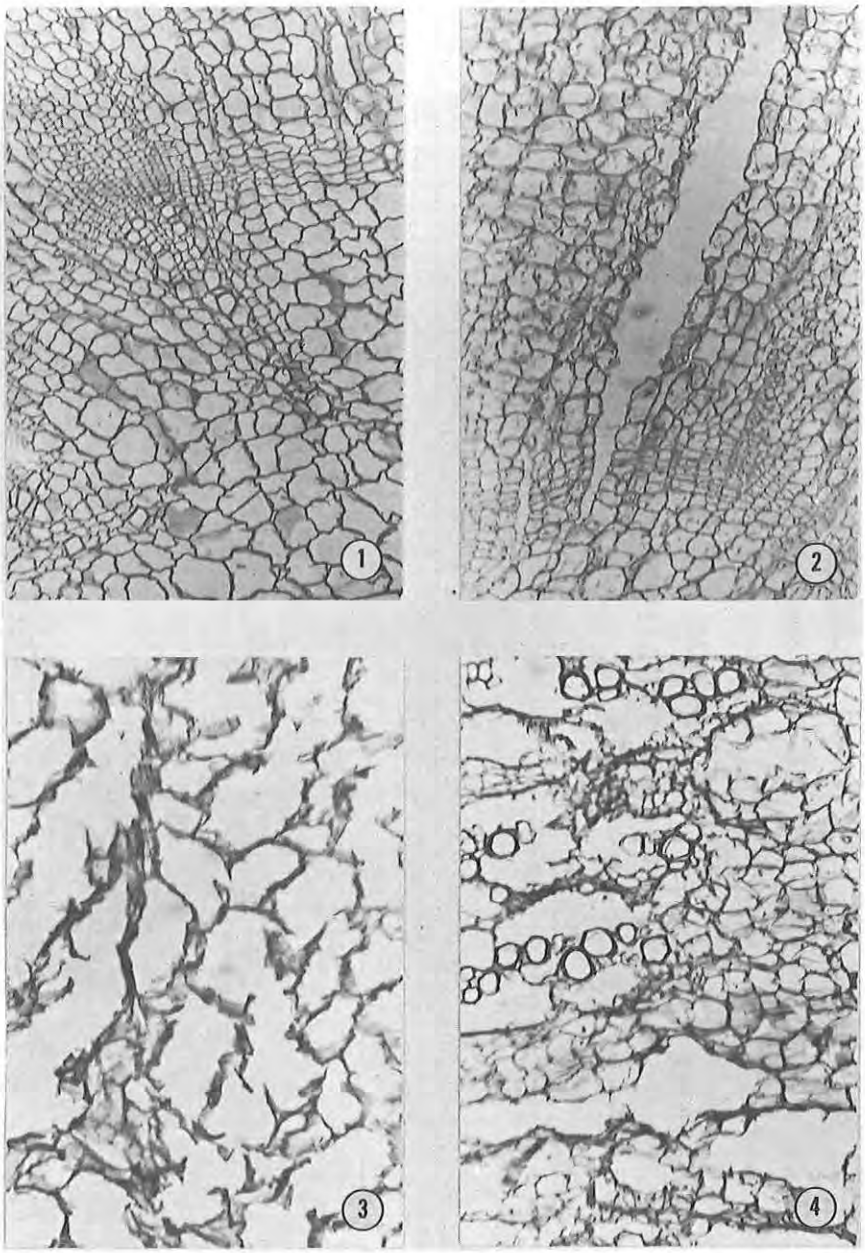


Photo 1: Carrot, raw, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 2: Carrot, cooked, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 3: Carrot, cooked, slowly frozen, freeze-dried, vacuum infiltrated with paraffin. Section thickness: 20μ. Magnification: X100.

Photo 4: Carrot, cooked, rapidly frozen, freeze-dried, vacuum infiltrated with paraffin. Section thickness: 20μ. Magnification: X100.

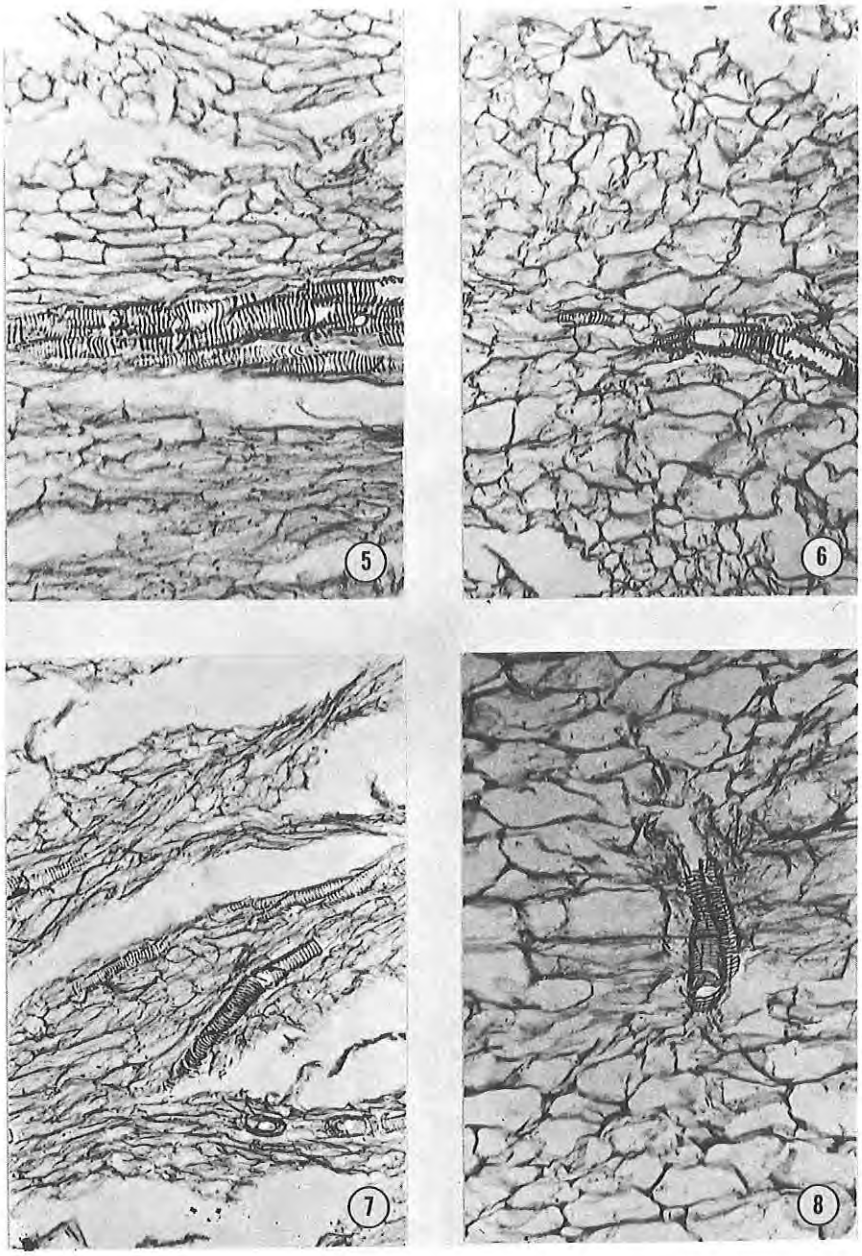


Photo 5: Carrot, cooked, slowly frozen, freeze-dried, equilibrated to 55% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 6: Carrot, cooked, rapidly frozen, freeze-dried, equilibrated to 55% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 7: Carrot, cooked, slowly frozen, freeze-dried, equilibrated to 70% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 8: Carrot, cooked, rapidly frozen, freeze-dried, equilibrated to 70% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

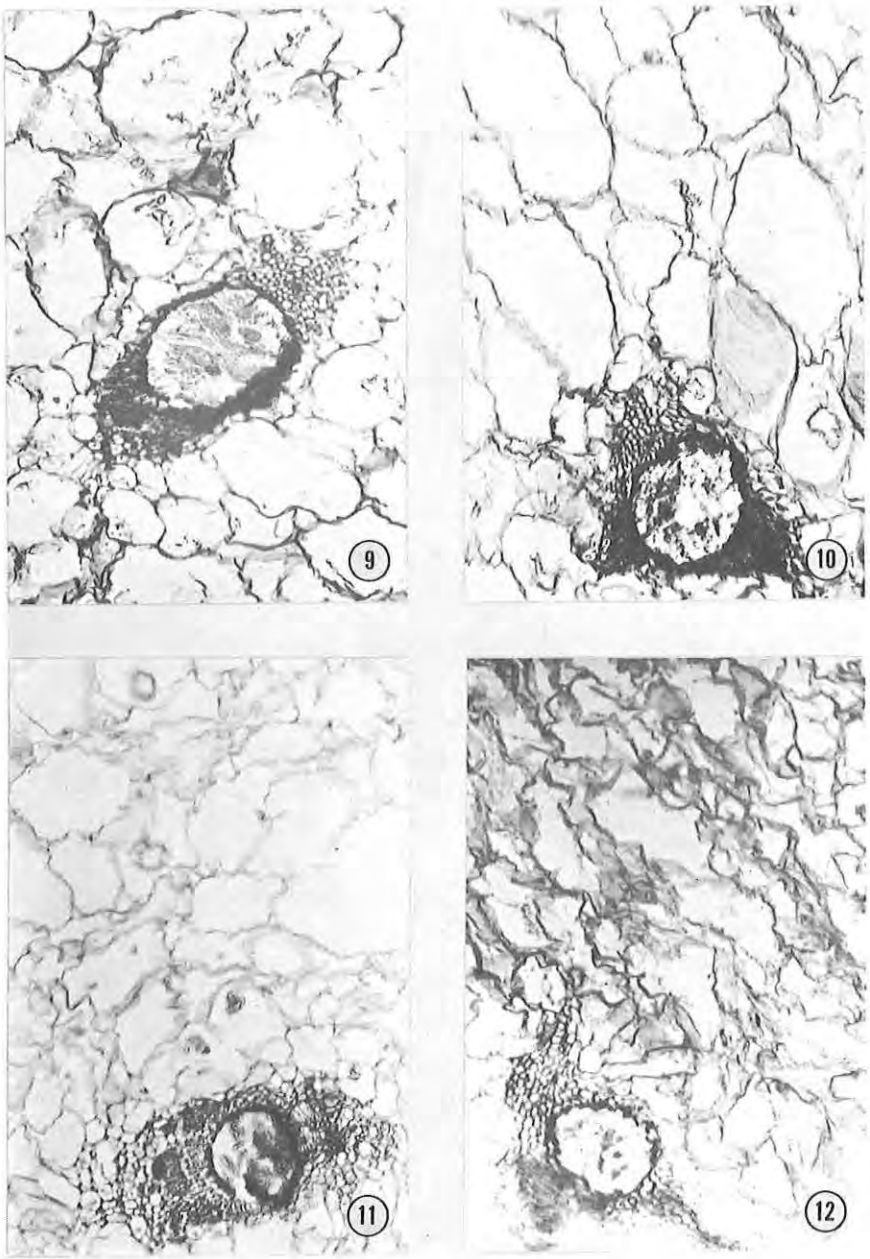


Photo 9: Peach, slowly frozen, thawed, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 10: Peach, slowly frozen, freeze-dried, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 11: Peach, slowly frozen, freeze-dried, compressed to 50% of original volume, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 12: Peach, slowly frozen, freeze-dried, compressed to 25% of original volume, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

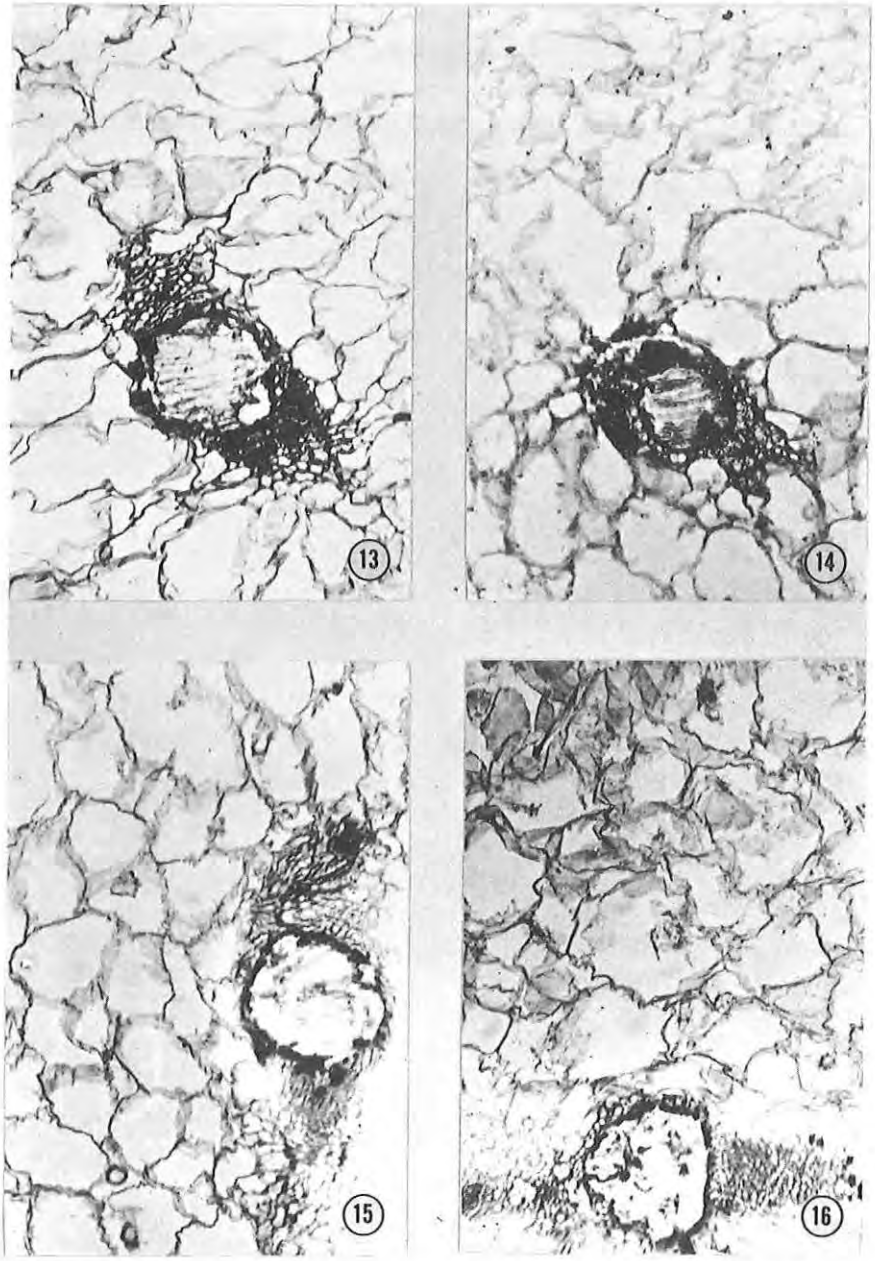
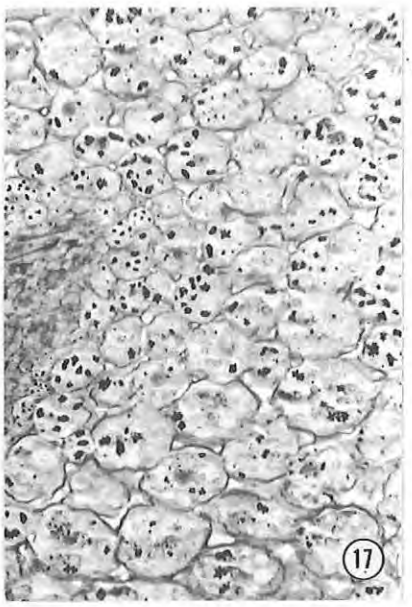


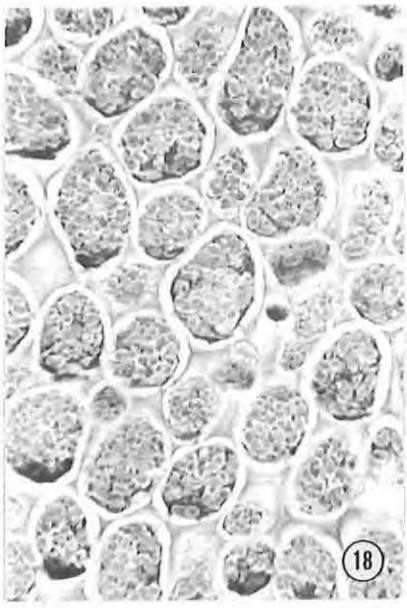
Photo 13: Peach, rapidly frozen, thawed, fixed, etc. Section thickness: 15μ. Magnification: X100.

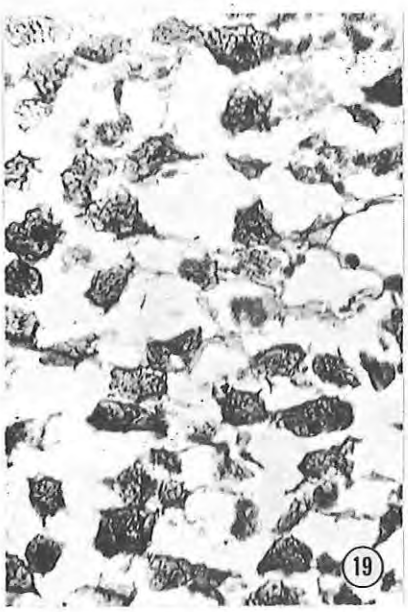
Photo 14: Peach, rapidly frozen, freeze-dried, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 15: Peach, rapidly frozen, freeze-dried, compressed to 50% of original volume, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 16: Peach, rapidly frozen, freeze-dried, compressed to 25% of original volume, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.







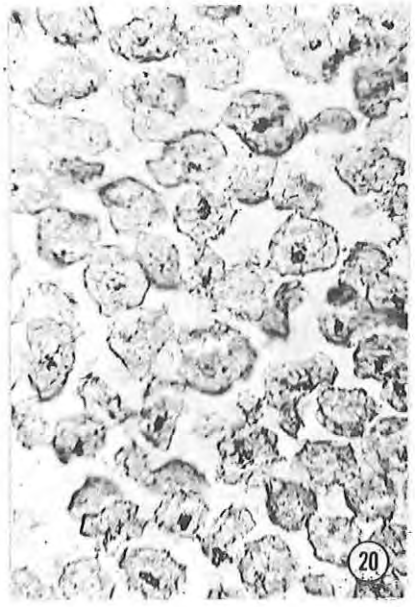


Photo 17: Pea, raw, fixed, etc. Section thickness: 15µ. Magnification: X100.

Photo 18: Pea, cooked, fixed, etc. Section thickness: 15µ. Magnification: X100.

Photo 19: Pea, cooked, slowly frozen, freeze-dried, vacuum infiltrated with paraffin. Section thickness: 20µ. Magnification: X100.

Photo 20: Pea, cooked, rapidly frozen, freeze-dried, vacuum infiltrated with paraffin. Section thickness: 20µ. Magnification: X100.

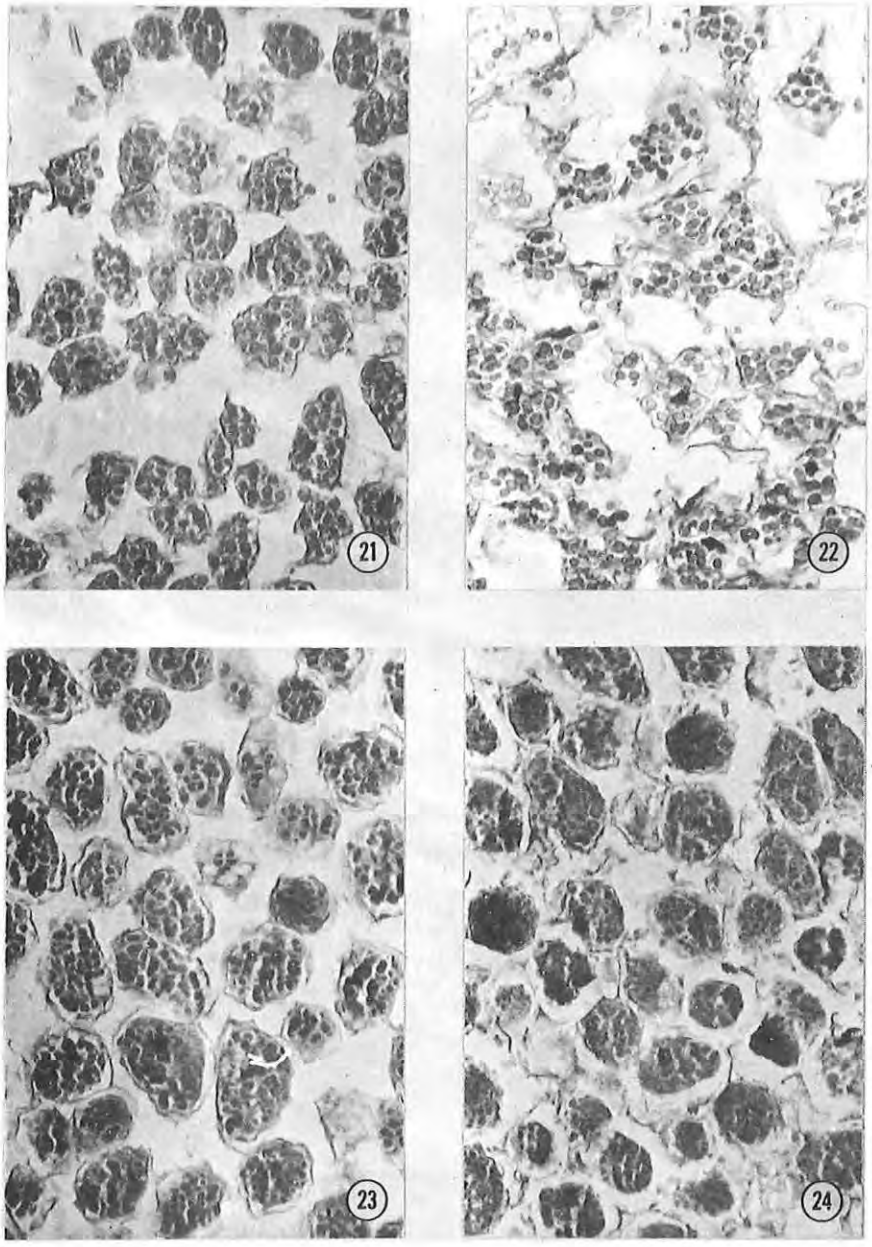
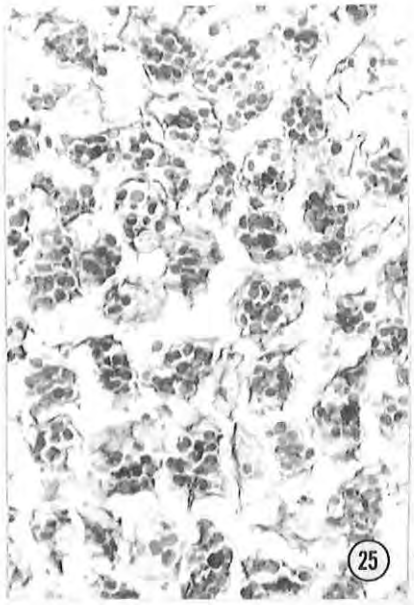


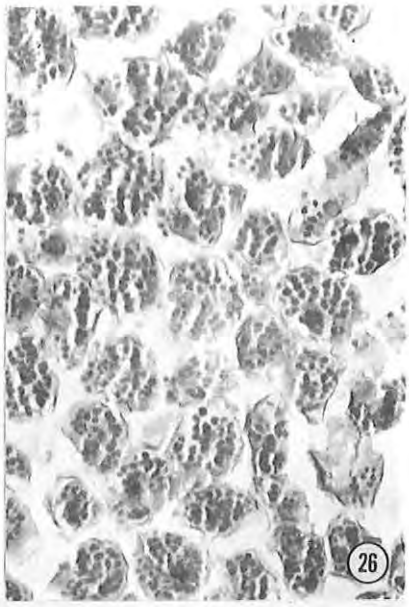
Photo 21: Pea, cooked, slowly frozen, freeze-dried, equilibrated to 70% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

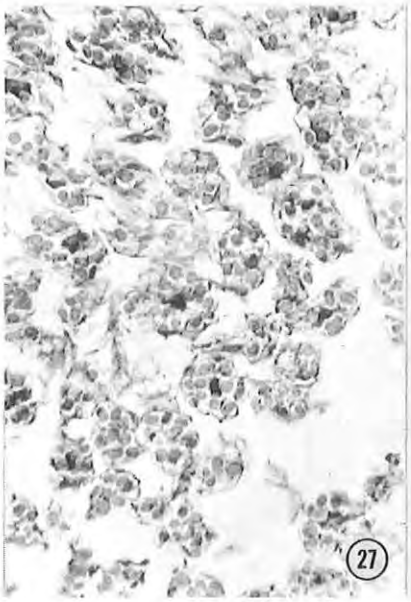
Photo 22: Pea, cooked, slowly frozen, freeze-dried, equilibrated to 70% R.H., compressed (2000 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 23: Pea, cooked, slowly frozen, freeze-dried, equilibrated to 80% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 24: Pea, cooked, slowly frozen, freeze-dried, equilibrated to 80% R.H., compressed (2000 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.







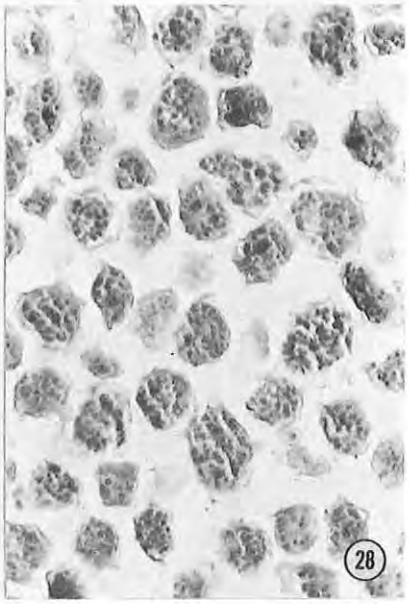
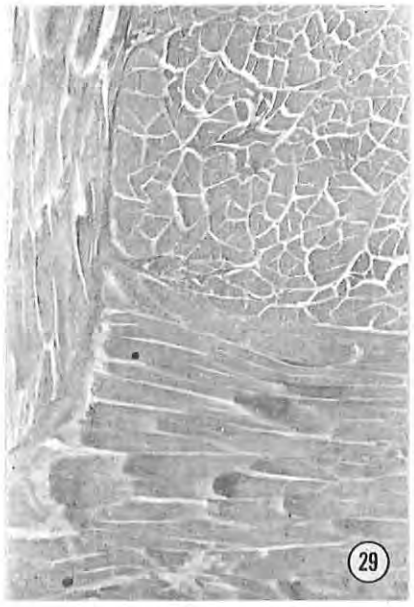


Photo 25: Pea, cooked, rapidly frozen, freeze-dried, equilibrated to 70% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 26: Pea, cooked, rapidly frozen, freeze-dried, equilibrated to 70% R.H., compressed (2000 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 27: Pea, cooked, rapidly frozen, freeze-dried, equilibrated to 80% R.H., compressed (300 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 28: Pea, cooked, rapidly frozen, freeze-dried, equilibrated to 80% R.H., compressed (2000 p.s.i.), rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.



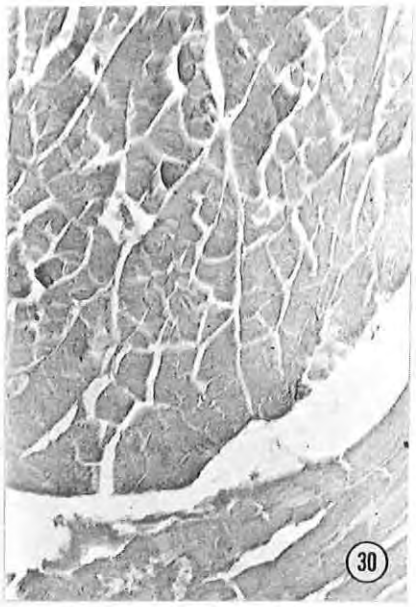


Photo 29: Shrimp, raw, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 30: Shrimp, cooked, slowly frozen, freeze-dried, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 31: Shrimp, cooked, slowly frozen, freeze-dried, compressed to 25% of original volume, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

Photo 32: Shrimp, cooked, slowly frozen, freeze-dried, compressed to 15% of original volume, rehydrated, fixed, etc. Section thickness: 15μ. Magnification: X100.

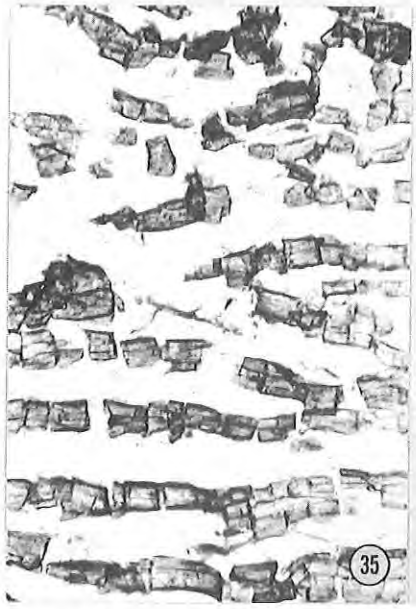


Photo 33: Shrimp, cooked, slowly frozen, freeze-dried, vacuum infiltrated with paraffin. Section thickness: 15µ. Magnification: X100.

Photo 34: Shrimp, cooked, slowly frozen, freeze-dried, compressed to 50% of original volume, vacuum infiltrated with paraffin. Section thickness: 15µ. Magnification: X100.

Photo 35: Shrimp, cooked, slowly frozen, freeze-dried, compressed to 25% of original volume, vacuum infiltrated with paraffin. Section thickness: 15µ. Magnification: X100.

Photo 36: Shrimp, cooked, slowly frozen, freeze-dried, compressed to 15% of original volume, vacuum infiltrated with paraffin. Section thickness: 15µ. Magnification: X100.

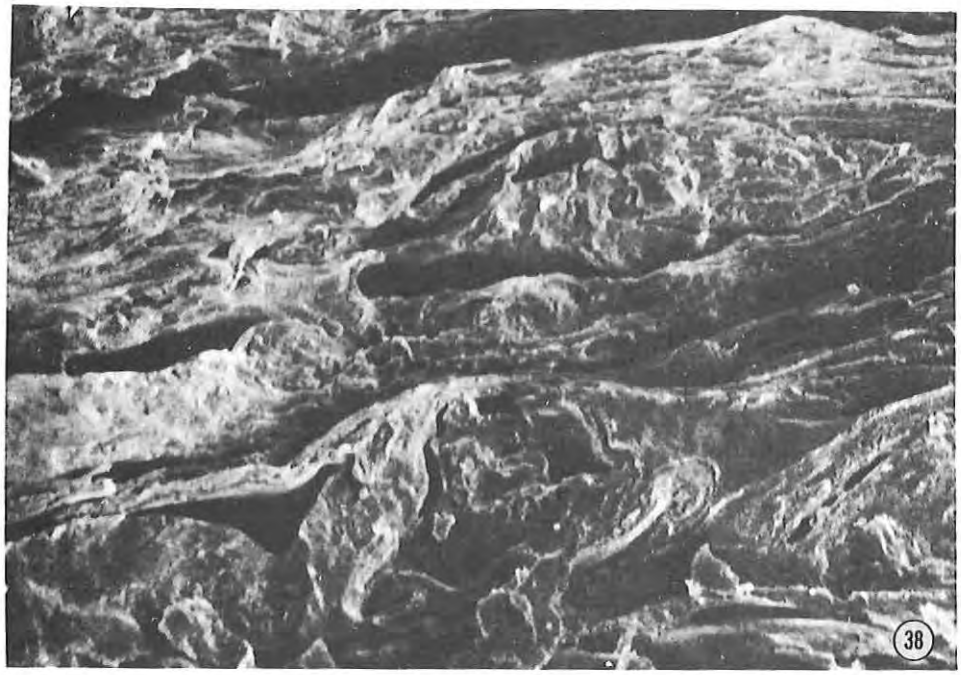
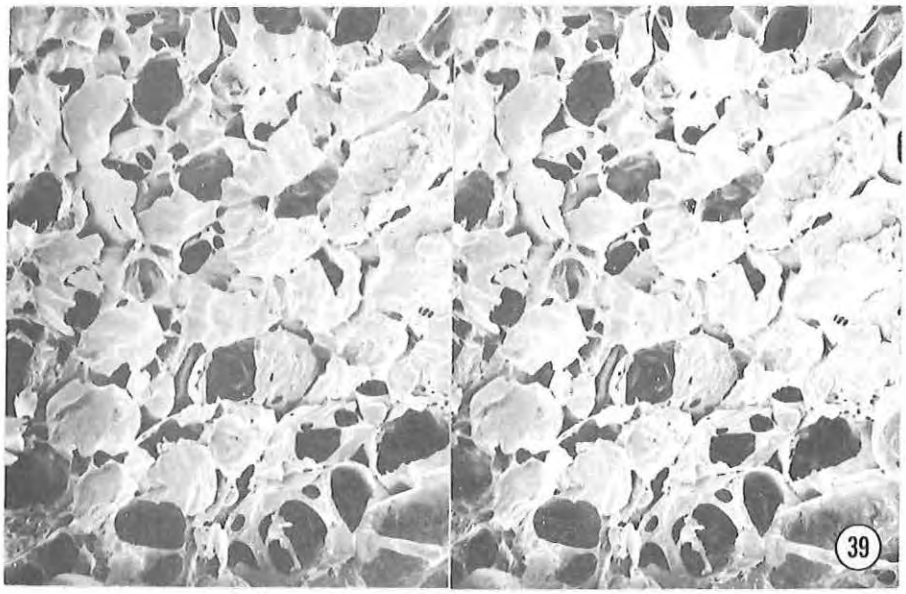


Photo 37: Carrot, cooked, frozen in air at -40°C, freeze-dried "at room temperature," remoistened (60% R.H., 25°C), compressed, cooled to -196°C, cleaved, warmed to 25°C and dried. View of the fracture surface. Magnification: X240.

Photo 38: Carrot, cooked, frozen in air at -40°C, freeze-dried "at room temperature," remoistened (60% R.H., 25°C), compressed, cooled to -196°C, cleaved, warmed to 25°C and dried. View of the fracture surface. Magnification: X2,200.



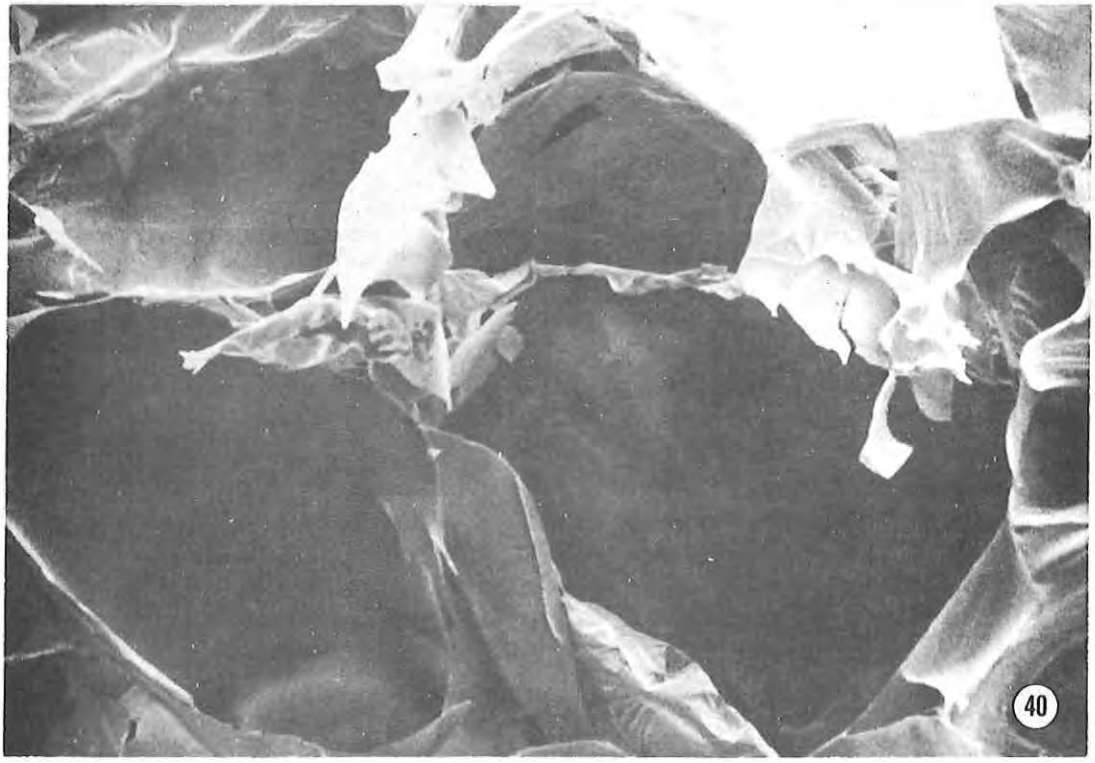
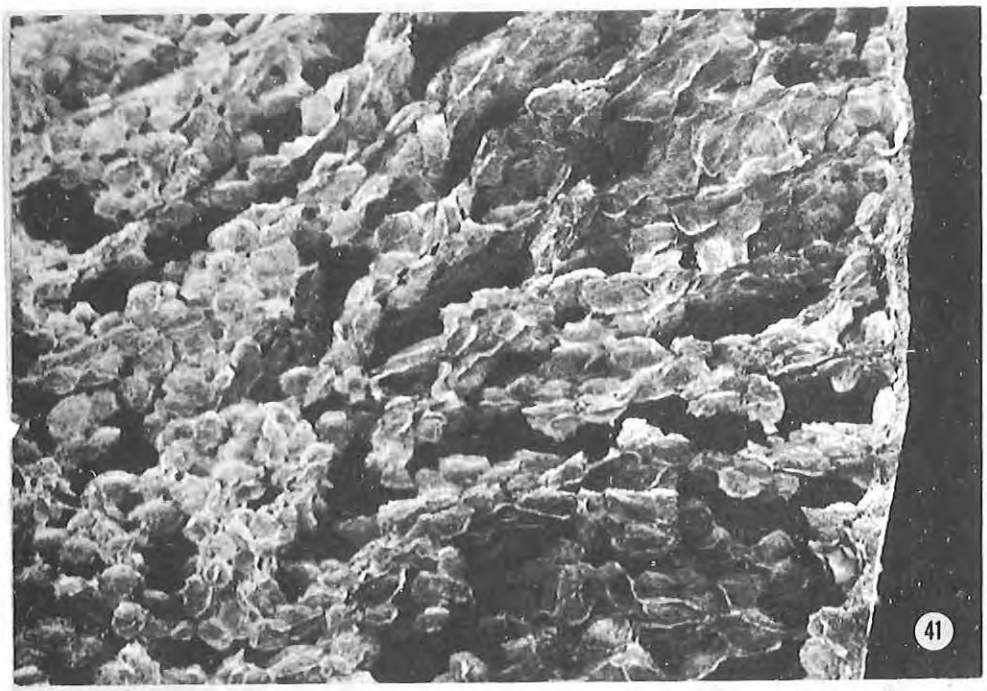


Photo 39: Peach, raw, frozen in air at -40°C, cooled to -196°C, cleaved and freeze-dried at -40°C. Stereo photographs of the fracture surface. Magnification: X100.

Photo 40: Peach, raw, frozen in air at -40°C, cooled to -196°C, cleaved and freeze-dried at -40°C. View of the fracture surface. Magnification: X550.



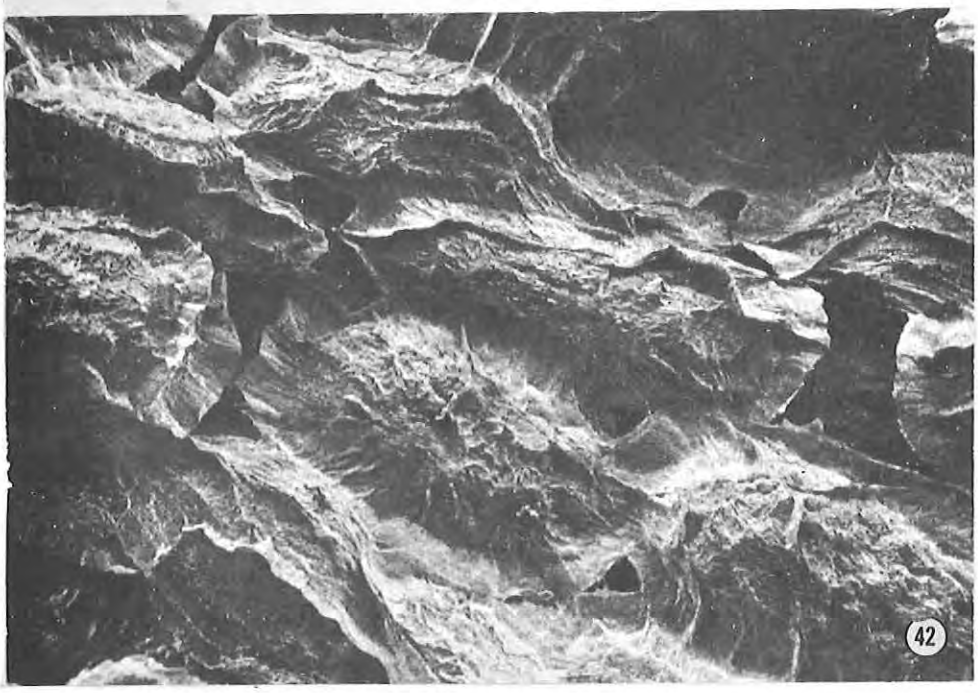
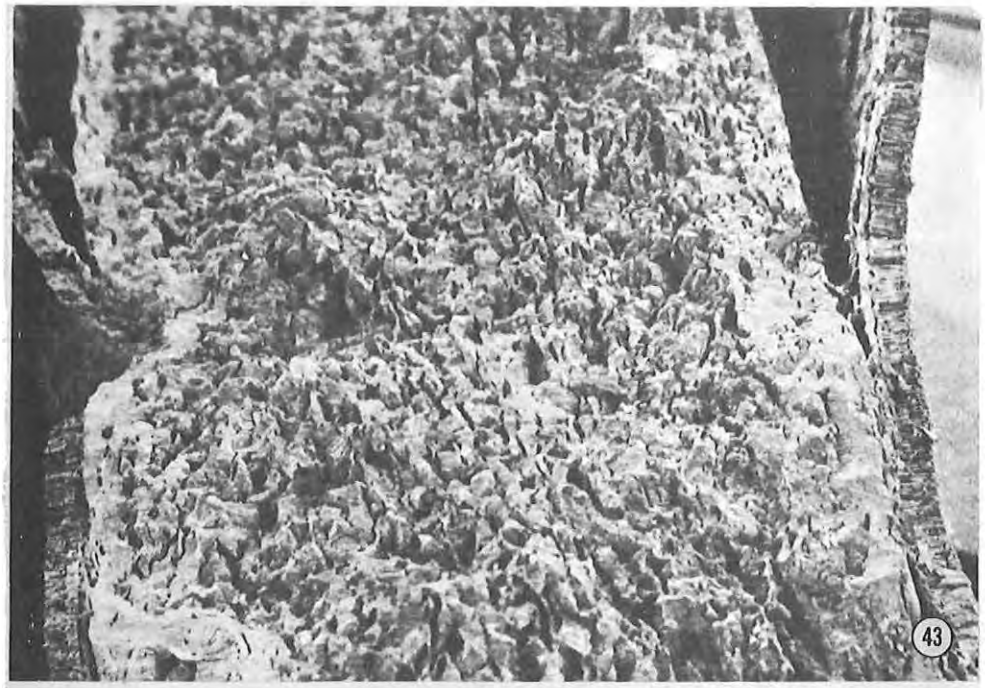


Photo 41: Pea, cooked, frozen in air at -40°C, cooled to -196°C, cleaved and freeze-dried at -40°C. Survey view of the fracture surface. Magnification: X100.

Photo 42: Pea, cooked, frozen in air at -40°C, cooled to -196°C, cleaved and freeze-dried at -40°C. View of the fracture surface. Magnification: X570.



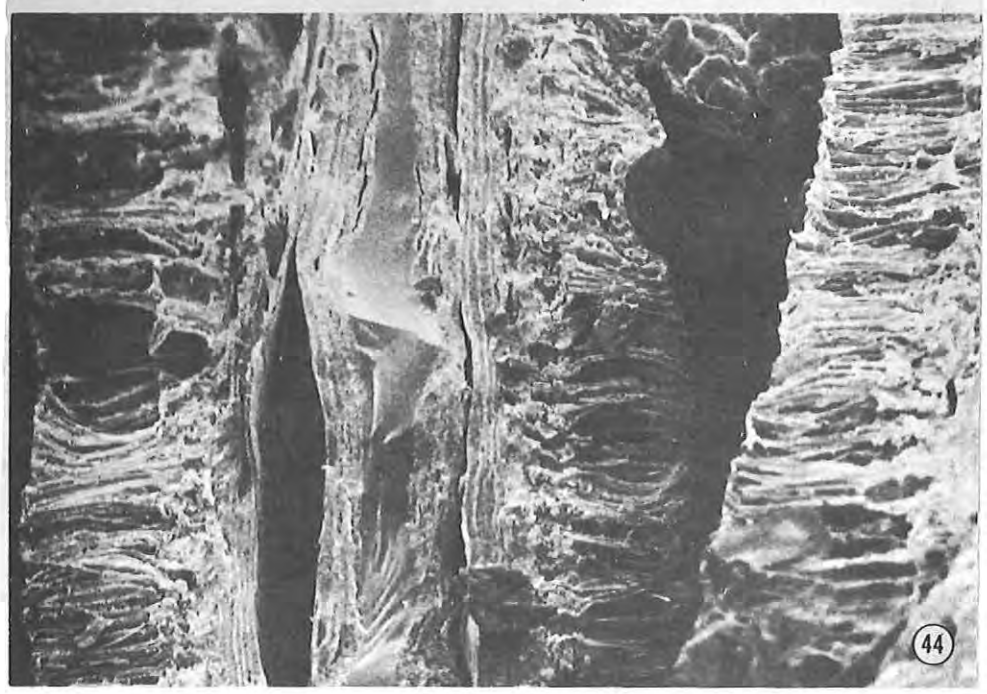
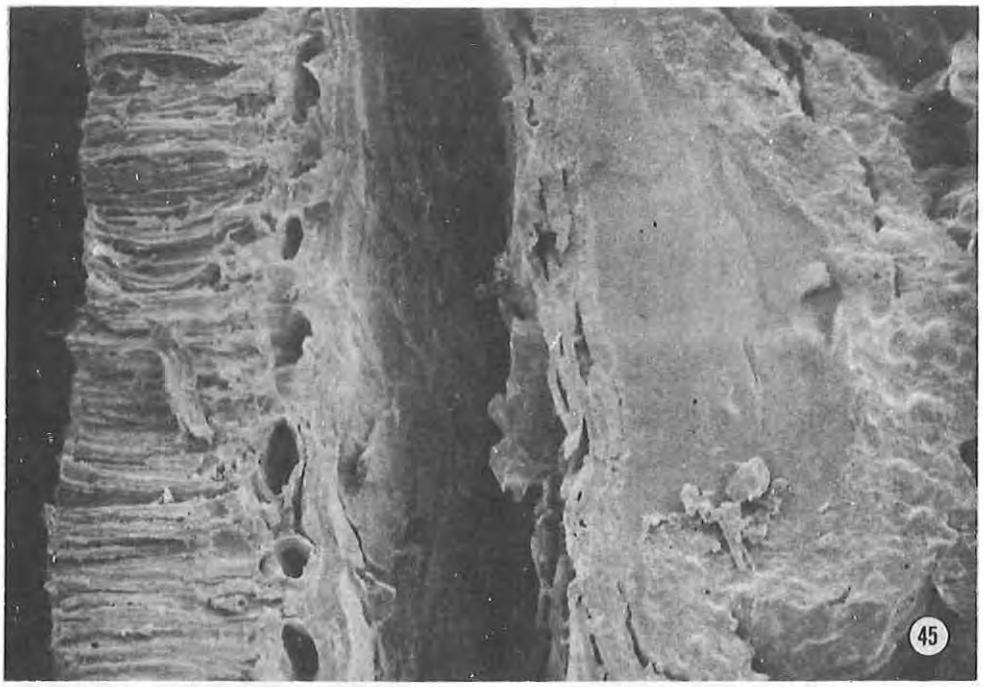
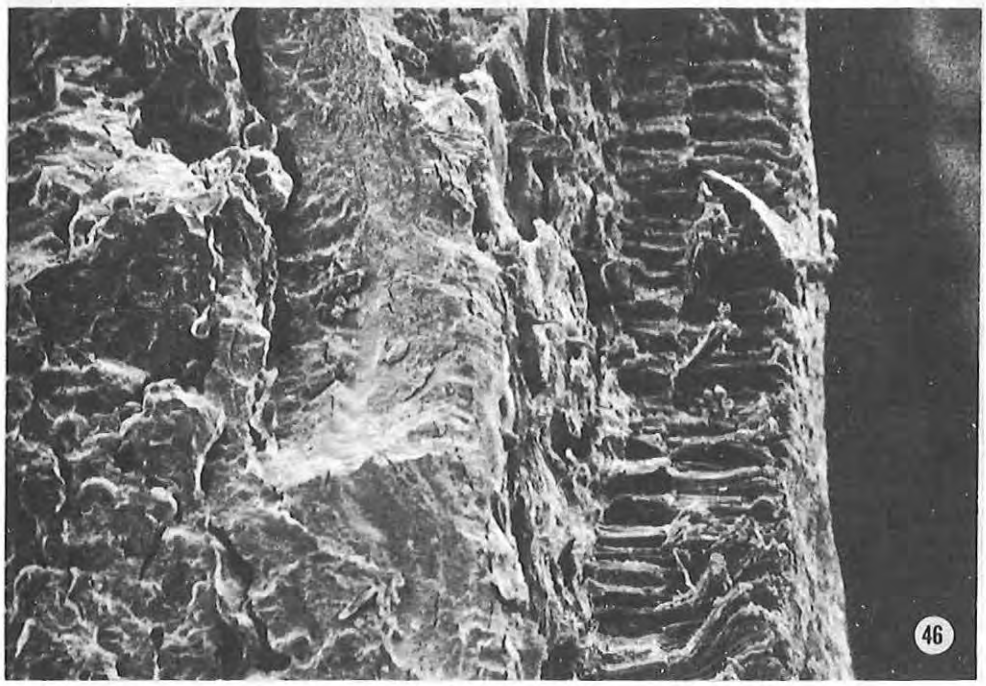


Photo 43: Pea, cooked, frozen in air at -40°C, freeze-dried "at room temperature," remoistened (70% R.H., 25°C), compressed, cooled to -196°C, cleaved, warmed to 25°C and dried. Survey photomicrograph. Magnification: X100.

Photo 44: Pea, cooked, frozen in air at -40°C, freeze-dried "at room temperature," remoistened (70% R.H., 25°C), compressed, cooled to -196°C, cleaved, warmed to 25°C and dried. View of the region adjacent to the hypocotyl. Magnification: X600.





Photos 45 & 46: Pea, cooked, frozen in air at -40°C, freeze-dried "at room temperature," remoistened (70% R.H., 25°C), compressed, cooled to -196 C, cleaved, warmed to 25°C and dried. Views of the exocarp and adjacent materials. Magnification: X570.

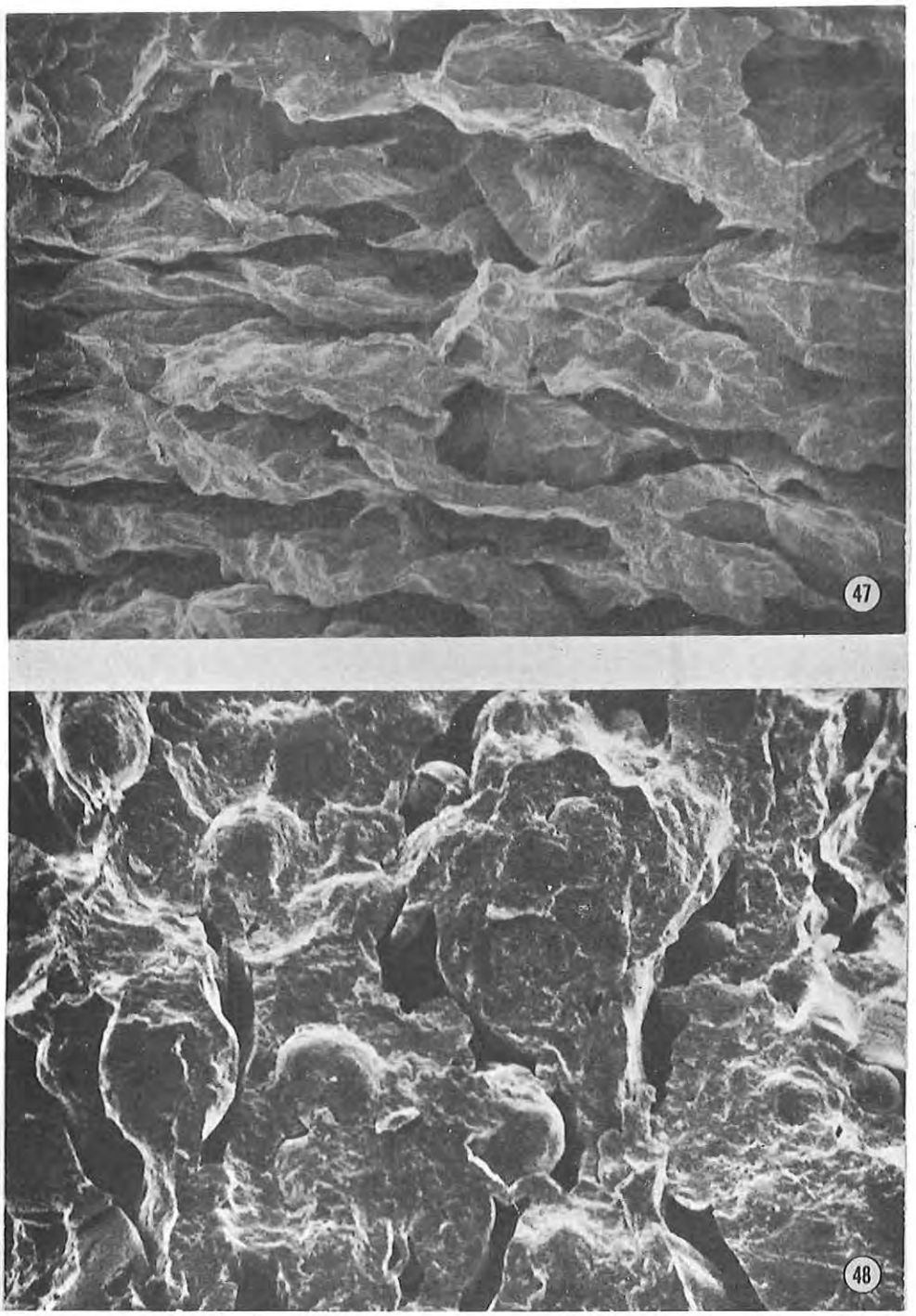


Photo 47: Pea, cooked, frozen in air at -40°C, freeze-dried "at room temperature," remoistened (70% R.H., 25°C), compressed, cooled to -196°C, cleaved, warmed to 25°C and dried. Endocarp, showing void spaces. Magnification: X600.

Photo 48: Pea, cooked, frozen in air at -40°C, freeze-dried "at room temperature," remoistened (70% R.H., 25°C), compressed, cooled to -196°C, cleaved, warmed to 25°C and dried. Endocarp, showing void spaces and effects of compression on individual cells —— starch grains appear here and there to have burst through cell walls. Magnification: X1,980.

REFERENCES

Ginnette, L. F. (1966), Technical Report 66-34-FD, U.S. Army Natick Laboratories, Natick, Mass.

Jensen, W. A. (1962), Botanical Histochemistry, W. H. Freeman and Company, San Francisco, Calif., pp. 90-94.

Lampi, R. A., et al. (1965), Technical Report FD-9, U.S. Army Natick Laboratories, Natick, Mass.

Luyet, B., and G. Rapatz (1957), Biodynamica, 7, 337-345.

Luyet, B. J., and A. P. MacKenzie (1967), Technical Report 67-90-FL, U.S. Army Natick Laboratories, Natick, Mass., pp. 19-24.

MacKenzie, A. P., and B. J. Luyet (1967), Cryobiology, 3, 341-344.

Salwin, H. (1962), in Freeze-Drying of Foods (Ed. F. R. Fisher), National Academy of Sciences - National Research Council, Washington, D. C., pp. 58-74.

Sass, J. E. (1964), Botanical Microtechnique, Iowa State University Press, Ames, Iowa, pp. 55-77.

FOOD LABORATORY DISTRIBUTION LIST

Copies

1 - Commanding General
US Army Medical Research & Development Command
Main Navy Building
Washington, D. C. 20315

2 - Commanding General
US Army Test & Evaluation Command
ATTN: AMSTE-BC
Aberdeen Proving Ground
Maryland 21005

1 - Commanding General
US Army Combat Development Command
Combat Service Support Group
Fort Lee, Virginia 23801

1 - Commanding Officer
US Army Medical Nutrition Laboratory
Fitzsimons General Hospital
Denver, Colorado 80240

1 - Commanding Officer
Edgewood Arsenal
ATTN: SMUEA-TSTI-TL
Edgewood Arsenal,
Maryland 21010

1 - Commander
Defense Personnel Support Center
ATTN: Directorate of Subsistence, DPSC-STS
2800 South 20th Street
Philadelphia, Pennsylvania 19101

1 - Executive Director
Joint Committee on Atomic Energy
Congress of the United States
Washington, D. C. 20510

Copies

1 - Commanding General
US Army Combat Development Command
ATTN: CDCMR-O
Fort Belvoir, Virginia 22060

1 - Commanding General
US Army Materiel Command
ATTN: AMCRD-JI
Department of the Army
Washington, D. C. 20315

1 - Commanding Officer
US Army Combat Development Command
Supply Agency
ATTN: CDCSA-R
Fort Lee, Virginia 23801

1 - Commanding Officer
US Army Arctic Test Center
ATTN: STEAC-TA
APO Seattle, Washington 98733

2 - Department of the Army
Headquarters, Fort Detrick
ATTN: Documents, Technical Library
Frederick, Maryland 21701

1 - Commandant of the Marine Corps
Code A04D
Washington, D.C.20380

1 - Commandant of the Marine Corps
Code CDE
Washington, D.C. 20380

Copies

1 - Stimson Library
ATTN: Documents Librarian
US Army Medical Field Service School
Brooke Army Medical Center
Fort Sam Houston, Texas 78234

1 - Director
Division of Biology & Medicine
US Atomic Energy Commission
Washington, D. C.20545

2 - Director
Biological Sciences Division
Office of Naval Research
Department of the Navy
Washington, D. C. 20360

3 - Office of the Coordination of Research
University of Rhode Island
Kingston, Rhode Island 02881

2 - National Aeronautics & Space Administration
ATTN: Acquisition Branch
'S-AK/DL'
PO Box 33
College Park, Maryland 20740

1 - US Department of Agriculture
Division of Acquisitions
National Agriculture Library
Washington, D. C. 20250

Copies

1 - Arctic Medical Research Laboratory, Alaska
ATTN: Librarian
APO Seattle, Washington 98731

2 - Quartermaster School Library
US Army Quartermaster School
Fort Lee, Virginia 23801

1 - US Naval Research Laboratory
Code 6140
Washington, D. C. 20390

4 - Exchange & Gift Division
Library of Congress
Washington, D. C. 20540

1 - Director
Division of Isotopes Development
US Atomic Energy Commission
Washington, D. C. 20545

2 - Director
Development Center
Marine Corps Development & Education Command
ATTN: Combat Service Support Division
Quantico, Virginia

2 - Headquarters 12th Support Brigade
ACofS Services
ATTN: Food advisor
Fort Bragg, North Carolina 28307

1 - Director
US Army Advanced Materiel Concepts Agency
Washington, D. C. 20315

Copies

1 - Headquarters, USAF
(AFRDDG)
DCS/Research &
Development
Washington, D.C. 20330

1 - National Aeronautics &
Space Administration
Ames Research Center
ATTN: J.E. Greenleaf,
239-4A
Moffett Field, Cali-
fornia 94035

2 - US Army Research Office
ATTN: Technical Library
3045 Columbia Pike
Arlington, Virginia
22204

1 - Chief, Life Sciences
Division
Army Research Office
Office of Chief of Researh
& Development
Washington, D.C. 20310

1 - Dr. Herbert E. Hall
Chief, Food Microbiology
National Center for Urban
& Industrial Health
Food Protection Research
222 East Central Parkway
Cincinnati, Ohio 45202

2 - Chief, Radiation Branch
Food Industries Division,
552
Business & Defense Service
Administration
US Department of Commerce
Washington, D.C. 20230

1 - US Atomic Energy Commission
Reports Section, Head-
Quarters Library
Main Station J-004
Division of Technical
Information
Washington, D.C. 20545

Copies

1 - Dr. Delbert M. Doty
Technical Director
Fats & Proteins Research
Foundation, Incorporated
3150 Des Plaines Avenue
Des Plaines, Illinois
60018

1 - Dr. A. W. Brant
Department of Food
Science & Technology
209 Roadhouse Hall
University of
California
Davis, California 95616

1 - Dr. William M. Roberts
Professor & Head
Department of Food
Science
North Carolina State
University
Raleigh, North Carolina
27607

1 - Mr. George Crapple
Technical Division
Wilson & Company
4200 South Marshfield
Chicago, Illinois 60609

1 - Library Southern Utili-
zation Research &
Development Division
Agricultural Research
Service
US Department of Agri-
culture
PO Box 19687
New Orleans, Louisiana
70119

1 - Mr. Harry W. Ketchum
Director, Radiation
Program
Food Industries Division,
BDSA
US Department of
Commerce, Room 4042
14th & Constitution Avs,NW
Washington, D.C. 20230

Copies

2 - Technical Library
USACDC Institute of
Land Combat
301 Taylor Drive
Alexandria, Virginia
22314

1 - Dr. Arthur Veis
Department of Medi-
cine & Biochemistry
Northwestern Univ.
301 East Chicago Ave.
Chicago, Ill. 60611

1 - Dr. H. D. Naumann
Department of Animal
Husbandry
University of Missouri
Columbia, Missouri
65202

1 - Dr. Philip K. Bates
363 17th Street
Santa Monica, Cali-
fornia 90402

1 - Dr. William J.Stadelman
Department of Animal
Science
Purdue University
Lafayette, Indiana 47907

1 - Dr. B. F. Buchanan
General Foods Technical
Center
555 South Broadway
Tarrytown, New York
10591

1 - Dr. Robert C. Baker
Department of Poultry
Husbandry
Cornell University
Ithaca, New York 14850

Copies

1 - Dr. Irving Pflug
Environmental Health
1112 Mayo Memorial
University of Minnesota
Minneapolis, Minnesota
55455

1 - Dr. Daniel Melnick
Division of Research
& Quality Control
Corn Products Company
Bayonne, New Jersey
07002

1 - Dr. K. G. Weckel
Department of Dairy &
Food Industry
Babcock Hall
University of Wisconsin
Madison, Wisconsin 53706

1 - Mr. Robert P. Dudley
Division of Research
& Development
George A. Hormel &
Company
Austin, Minnesota 55912

1 - Dr. A. M. Pearson
Department of Food
Science
Michigan State Univ.
East Lansing, Michigan
48823

1 - Dr. Walter O. Lundberg
The Hormel Institute
Austin, Minnesota 55921

1 - Mr. Frank K. Lawler
Editor
Food Engineering
Chestnut & 56th Streets
Philadelphia, Pennsyl-
vania 19133

Copies

1 - Dr. Harold S. Olcott
Professor
Marine Food Science & Technology
10 Hilgard Hall
University of California
Berkeley, California 94720

1 - Dr. Owen Fennema
Department of Food Science & Industries
University of Wisconsin
Madison, Wisconsin 53706

1 - Professor Betty M. Watts
Department of Food & Nutrition
Florida State University
Tallahassee, Florida 32306

1 - Dr. Floyd Olsen
Associate Director for Research
Oscar Mayer & Company
Madison, Wisconsin 53701

1 - Dr. A. Barde Rogers
Research Laboratories
Armour & Company
Oak Brook, Illinois 60522

1 - Mr. W. R. Schack
Swift & Company
Research & Development Laboratories
Oak Brook, Illinois 60521

1 - Evans Research & Development Corporation
250 East 43rd Street
New York, New York 10017

Copies

1 - Professor V.H.Nielsen
Department of Dairy & Food Industry
Iowa State University
Ames, Iowa

1 - Dr. Kenneth N. May
Poultry Department
University of Georgia
Athens, Georgia 30601

1 - Dr. Alan P. MacKenzie
American Foundation for Biological Research
RFD 5, Box 137
Madison, Wisconsin 53716

1 - Mr. Darwin Kueck
Research & Development Laboratories
Rath Packing Company
Waterloo, Iowa 50704

1 - Mr. Robert M. Weiss
Research & Development Laboratories
The Pillsbury Company
311 Second Street, SE
Minneapolis, Minnesota 55414

1 - Dr. Norman G. Roth
Whirlpool Corporation
300 Broad Street
St. Joseph, Michigan 49085

1 - Dr. Marcus Karel
Department of Nutrition & Food Science
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139

Copies

1 - Mr. O. B. Gerrish
Midwest Research Institute
425 Volker Boulevard
Kansas City, Missouri
64110

1 - Dr. William W. Marion
Department of Poultry Science
Iowa State University
Ames, Iowa 50010

1 - Dr. Robert Cassens
Department of Meat & Animal Science
University of Wisconsin
Madison, Wisconsin
53706

1 - Professor Maurice W. Hoover
Department of Food Science
North Carolina State University
Raleigh, North Carolina
27607

1 - Mr. F. Warren Tauber
Food Products Division
Union Carbide Corporation
6733 West 65th Street
Chicago, Illinois 60638

1 - Dr. Paul A. Lachance
Department of Food Science
Rutgers University
New Brunswick, New Jersey 08903

1 - Professor A.I. Nelson
Department of Food Science
University of Illinois
Urbana, Illinois 61803

Copies

1 - Dr. Morton Cole
Archer Daniels Midland Company
10701 Lyndale Avenue, South
Bloomington, Minnesota
55440

1 - Dr. C. O. Chichester
Department of Food Science
University of California
Davis, California 95616

1 - Mr. Norman Ishler
Tronchemics Research Incorporated
480 US Route #46
South Hackensack, New Jersey

1 - Mr. William Sulzbacher
Chief
Meat Laboratory
US DA - ARS
Beltsville, Maryland
20705

1 - Dr. Roy E. Morse, V.P., Research
Pepsico Incorporated
500 Park Avenue
New York, New York
10022

1 - Dr. Amihad Kramer
University of Maryland
Department of Horticulture
College Park, Maryland
20742

1 - Dr. George Mountney
Department of Poultry Science
Ohio State University
674 West Lane Avenue
Columbus, Ohio 43210

Copies

1 - Dr. V. O. Wodicka
Technical Division
Hunt-Wesson Foods
1645 West Valencia Drive
Fullerton, California 92634

1 - Mr. Edward Seltzer
Assistant Director for Research
Thomas J. Lipton Incorporated
800 Sylvan Avenue
Englewood Cliffs, New Jersey 07632

1 - Dr. William B. Esselen, Head
Department of Food Science & Technology
University of Massachusetts
Amherst, Massachusetts 01002

Copies

1 - Dr. Hans Lineweaver
Chief
Poultry Laboratory, W.R L, USDA
Albany, California 94706

1 - Dr. H. H. Litchfield
Chief Biochemistry
Battelle Memorial Institute
505 King Avenue
Columbus, Ohio 43201

FOOD LABORATORY INTERNAL DISTRIBUTION LIST

Copies

25 - Chief, Technical Plans Office, NLABS
(20 for transmittal to Defense Documentation Center)

2 - Technical Library, NLABS

10 - Program Coordination Office, Food Laboratory, NLABS

7 - Division Chiefs, Food Laboratory, NLABS

2 - Marine Liaison Officer, NLABS

5 - Air Force Liaison Officer, NLABS

1 - Director, Earth Sciences Laboratory, NLABS

2 - Director, General Equipment and Packaging Laboratory, NLABS

3 - Director, Pioneering Research Laboratory, NLABS

1 - Commanding Officer, US Army Research Institute of Environmental Medicine, NLABS

25 - Project Officer and Alternate Project Officer, Food Laboratory, NLABS

Unclassified
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*

American Foundation for Biological Research
Madison, Wisconsin

2a. REPORT SECURITY CLASSIFICATION

Unclassified

2b. GROUP

3. REPORT TITLE

RECOVERY OF COMPRESSED DEHYDRATED FOODS

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

Final June 1967 - December 1968

5. AUTHOR(S) *(First name, middle initial, last name)*

A. P. MacKenzie and B. J. Luyet

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
July 1969	121	6

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
DAAG 17-67-C-0126	
b. PROJECT NO. 1M624101D553	
c.	9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*
d.	70-16-FL FL-90

10. DISTRIBUTION STATEMENT

This document has been approved for public release and sale; its distribution is unlimited.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	U.S. Army Natick Laboratories Natick, Massachusetts 01760

13. ABSTRACT

Seven foods were frozen at various rates, freeze-dried in different ways and adjusted to different moisture contents by exposure to atmospheres of controlled R.H. The resultant materials were compressed, dried, and tested for their capacities to recover initial form and quality on rehydration.

It was observed that processing conditions insuring best recovery can be defined in terms of relative humidity to which a food is exposed prior to compression.

Freeze-dried foods of predetermined moisture content were produced by newly developed processes involving only the sublimation of ice and the direct desorption of a part of the water remaining unfrozen. These methods offered effective alternatives to the method by which fully freeze-dried materials are moistened by resorption prior to compression.

Compression in vacuum was successfully demonstrated. Similarly, freeze-drying, adjustment of the water content, compression, and final drying were realized in a single apparatus. These methods were each shown to possess special advantages.

Additional experiments were conducted on the compression of solvent-extracted foods. Freeze-dried, compressed, and restored foods were also examined by light and electron microscopic techniques. From these additional studies some indications were obtained of the mechanisms and factors contributing to restoration.

DD FORM 1 NOV 68 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

Unclassified
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Rehydration	8,7		4			
Freeze dried foods	9,7		9			
Compressed foods	9,7		9			
Humidity	6					
Moisture content	6					
Vacuum	6					
Examination			8			
Electron microscopes			10			
Light (visible radiation)			10			